

JPL Contract 951709

STERILIZABLE LIQUID PROPULSION SYSTEM

Second Quarterly Progress Report

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April 1967

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STERILIZABLE LIQUID PROPULSION SYSTEM
244 SECOND QUARTERLY PROGRESS REPORT, 4 in MK 127

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FOREWORD

This document is the second issue of the Quarterly Progress Report and is submitted in accordance with Article 1(a)(1)(v)(E) and 2(b)(5) of JPL Contract 951709.

CONTENTS

	Page
Foreword	ii
Contents	iii
I. Introduction	1
II. Conclusions	2
III. Recommendations	4
IV. General Report	6
A. Rocket Engine Selection	6
B. System Design and Analysis	10
C. Component Design and Analysis	13
D. Material Investigations	26
E. Test Plan	26
F. Test Facilities	26
	thru
	30
Appendix --Materials Compatibility Study	A-1
	thru
	A-48
 <u>Figure</u>	
1 Artist's Concept of Planar Configuration	11
2 Sterilizable Liquid Propulsion System	12
3 Teflon Bladder	18
4 Screen Trap	20
 <u>Table</u>	
1 Nominal Performance and Design Parameters for the Available Engines	7
2 Final Engine Selection	9
3 Pressurant Gas Sphere Selection	14
4 Propellant Storage Sphere Selection	16
5 Expulsion Bladder Selection	17
6 Hand Shutoff Valve Selection	22
7 Gas Pressure Regulator Selection	23
8 Y4-in. Solenoid Valve Selection	24
9 Filter Selection	25

I. INTRODUCTION

This is the second quarterly progress report submitted in accordance with JPL Contract 951709. The report covers the period from 2 January 1967 thru 31 March 1967.

The program involves the exposure of an assembled and fueled bipropellant liquid propulsion system to the ethylene oxide (ETO) and heat sterilization environments specified by JPL specification VOL 50503 ETS. After exposure the system will be fired for 300 sec.

The program plan includes a design and component selection phase during which the propulsion system design is evolved. A second phase will involve the procurement of components for both a component test series and for assembly into the complete system. The third phase of the program, which is being carried on in parallel with the design phase, is a materials investigation. During this phase, data are being collected and testing is taking place. Where data do not exist, testing is being conducted to provide the necessary information. The fourth phase of the program involves the assembly and test of the complete propulsion system. The system will be assembled and propellants loaded and then exposed to ETO and heat sterilization cycles. No attempt will be made to sterilize or to verify sterilization. The intent is to prove the feasibility of exposing a loaded bipropellant propulsion system to both the ETO and heat sterilization environments without system degradation. This will be proved by a 300-sec hot firing of the system immediately after exposure to the environments. As a final verification the system will be disassembled and the component parts tested and inspected for degradation.

During this report period the basic system design was established and detailed structural design was initiated. The 600-hr material screening tests were completed, allowing final selection of components. Screening test results are presented as an appendix to this report. Component procurement orders were placed with suppliers. The test plan for both component and system testing was completed and was submitted to JPL for approval.

During the next period detailed design will continue and system structural fabrication will be initiated. Long-term propellant tests will be started using small-scale simulated propellant tanks. All materials present in the full-scale tanks will be present in the small-scale tanks.

The ETO and heat sterilization chamber will become operational and will be qualified.

II. CONCLUSIONS

The following conclusions were reached as a result of work during this reporting period:

- 1) The Marquardt **Model R-4D** engine should be selected since all materials used in its construction will withstand both decontamination and terminal sterilization cycles;
- 2) Propulsion system hardware that will contact propellants during sterilization cycles should be constructed of the following materials:
 - a) Propellant tanks -
 - (1) N_2O_4 - titanium (6Al-4V)
 - (2) MMH - titanium (6Al-4V), stainless steel (17-4), aluminum (2014-T6 or 2219-T8),
 - b) Plumbing lines -
 - (1) N_2O_4 - titanium (6Al-4V)
 - (2) MMH - stainless steel (304, 304L, 321, or 347), aluminum (2014, 2219, 2024, or 6061),
 - c) Valve bodies -
 - (1) N_2O_4 - titanium (6Al-4V) or aluminum with sulfuric or chromic acid anodize on all surfaces that will be in contact with propellant during sterilization
 - (2) MMH - aluminum (2014, 2024, or 6061) or stainless steel (304, 304L, 321, or 347),
 - d) Positive displacement devices for propellants -
 - (1) N_2O_4 - Teflon bladder (TFE-FEP laminate) or titanium sieves
 - (2) MMH - stainless steel screen (304 or equivalent) aluminum screen (6061 or 5056) or Teflon bladder (TFE-FEP laminate),

e) Burst discs -

- (1) N_2O_4 - titanium (6Al-4V)
 - (2) MMH - aluminum (1100 or 6061), or stainless steel (304, 321, or 347);
- 3) Propulsion system hardware that will not be exposed to propellants during contamination and sterilization cycles should be constructed of the materials listed in the literature survey (contained in the first Quarterly Report) as applicable to the particular design application;
 - 4) The ASI vapor detector for N_2O_4 is suitable for use in an ethylene oxide atmosphere. The MMH sensor will not function properly and should not be used;
 - 5) An automatic vent and purge system is required during decontamination since either propellant can react with the ethylene oxide. If there is a propellant leak, a serious pressure rise could occur inside the chamber causing rupture;
 - 6) The entire MMH system requires stringent cleaning, followed by passivation with propellant-water mixture to ensure that reaction of propellants with contamination does not occur after propellant loading. This is especially true for screens and welded joints that may entrap oxides or other reactive contaminants;
 - 7) Additional testing of effects of Freon 12 on titanium structures should be conducted. Information available, to date, is not conclusive.

III. RECOMMENDATIONS

As a result of work during this quarter, the following recommendations are made:

- 1) All stainless steels tested have sustained intergranular corrosion during the sterilization cycle in the presence of N_2O_4 . In addition, a viscous brown deposit has been formed in the propellant. It is recommended that the compounds formed be analyzed by infrared spectroscopy and X-ray diffraction with further characterization of the compounds by differential thermal calorimetry and analysis;
- 2) Depending on the results of 1) above, investigation of potential inhibitors should be studied and tested. It is anticipated that HO, HF, chromium ions and nitrates will be considered;
- 3) Current testing at sterilization temperatures indicates that inadequate cleaning and passivation of amine fuels such as MMH, UDMH, or hydrazine can result in decomposition. The decomposition can lead to very high pressures resulting in tank rupture, formation of corrosive compounds, or degradation of normal performance. A test program should be initiated to develop techniques of cleaning and passivation of representative materials and cavities simulating tankage, plumbing screens, bladders, and components. Cleaning methods should include chemical and mechanical methods, which may be augmented by ultrasonic techniques;
- 4) Results of testing of screen materials has revealed:
 - a) The large surface area of screens render them far more susceptible to attack than sheet metal,
 - b) There is limited screen availability in compatible materials,
 - c) Fine screens and foils must be given special handling and procedures during construction and fabrication.

It is recommended that a program be initiated to fabricate titanium and tantalum screens. In the case of titanium, emphasis should be placed on obtaining ductile wire by chem-milling techniques. While tantalum is quite ductile and available in wire form down to 0.003 in., we recommend a development program to extend this to weaving a mesh of at least 200 lines per inch;

- 5) It is recommended that alternative methods of oxidizer propellant sterilization be investigated. Two techniques of nonthermal sterilization should be considered -- ultraviolet radiation and filtration. Each technique should be evaluated by bio-assay procedures;
- 6) Teflon bladders have historically demonstrated a high permeability of propellants. Recently Teflon bladder material tests in N_2O_4 at 275°F have shown a tendency to slough off Teflon particles that may cause filters to clog or pulsing valves to leak. It is recommended that a development program be set up to deposit tantalum, columbium, and/or gold on Teflon laminate coupons and perform propellant compatibility tests and mechanical properties tests, such as abrasion, permeation, and adhesion;
- 7) Current propellant combinations all show undesirable characteristics at sterilization temperatures. Future propellants may possess more desirable characteristics such as lower vapor pressure, etc. It is recommended that materials prescreening tests be initiated to determine the reactions of such propellants as MON-10, IRFNA/55- N_2C_4 /45, MMH hydrate, and MAF-5 to sterilization requirements. Propellants that show favorable corrosion, decomposition, and vapor pressure properties should then be exposed to a more comprehensive evaluation program.

IV. GENERAL REPORT

A. ROCKET ENGINE SELECTION

At the end of the first phase of engine selection, five engines were still under consideration for use on this program. The engines were as follows:

- 1) Rocketdyne - Beryllium - heat sink;
- 2) Marquardt - Model R-4D - radiation;
- 3) Thiokol, RMD - Model C-1 - regenerative;
- 4) TRW Systems - MIRA-150R - radiation;
- 5) Bell Aerosystems - Model 8414 - radiation.

Engine manufacturers were contacted to determine engine availability and cost. In addition, detailed performance data and engine test histories were requested. As a result of these inquiries, the Rocketdyne and the Bell engines were eliminated. The three remaining engines were evaluated as to ETO and thermal compatibility. All are compatible with the defined sterilization system requirements. The final selection of the engine considering the ability of the three engines to meet the sterilization requirements is, therefore, based on previous test experience with the selected propellants, system adaptability, component simplicity, reliability, and development status.

Throttling of the rocket engine is not a requirement for the sterilization system and the additional components required make the engine more complex. Also, an engine with maximum test experience is desired to provide a good bench mark. The TRW MIRA-150R engine has been omitted for this reason, and because the high inlet pressure of 350 psi minimum, for this engine is much higher than necessary for this system. The vapor pressure of MMH at 275°F is 63 psia, therefore, the propellant tank pressure requirement was established to meet the minimum operating supply pressure of the available engines. The Marquardt and RMD nominal supply pressures are 180 psia and were used as the basis for a selected system propellant tank operating pressure of 260 ± 10 psia. Nominal performance and design parameters for the available engines are presented in Table 1.

Table 1 Nominal Performance and Design Parameters for the Available Engines

Parameters	Marquardt R-4D	RMD C-1	TRW MIRA-150R
Vacuum Thrust (lb_f)	100	100	100
Specific Impulse (sec) (vacuum nominal)	293	295	291
Characteristic Velocity (fps) [nominal (C*)]	5350	5295	5330
Thrust Coefficient (vacuum nominal)	1.70	1.80	1.76
Operating Life (sec) (demonstrated)	12,960	12,706	--
Maximum Steady-State Duration with NTO/MMH (sec)	1020	1400	--
Chamber Pressure (psia) (nominal)	97	93	--
Oxidizer, N_2O_4 to Fuel, MMH	1.6	1.6	1.6
Mixture Ratio (nominal)			
Nozzle Area Ratio, without Bell	6,7:1	5:1	
Nozzle Area Ratio	40:1	60:1 (overtuned bell)	33,7:1 (80% bell)
Throat Area ($in.^2$)	0.592	0.586	0.785
Characteristic Length (L^*)	11.2	10.7	18
Contraction Ratio	4,17:1	3,38:1	5,5:1
Injector Type	Multidouplet	Vortex	Coaxial sheet
Surge Pressure Capability (psi)	1000	480	1000
Valve Inlet Pressures, Nominal Supply (psi)			
Fuel	180	178-191	350-750
Oxidizer	180	178-191	350-750
Overall Length (in.)	13.4	17.29	17.5
Maximum Diameter (in.)	6.5	6.75	8.2
Thrust Mount	Flange Injector End	Flange Injector End	Engine Mount Bracket

The engine selection criteria for final screening between the Marquardt R-4D and RMD C-1 engines are presented in Table 2. The engines were rated to provide the basis of final selection indicated in Table 2.

The Marquardt engine was selected based on test experience of the fixed R-4D design. The RMD C-1 engine has more desirable design features, but lacks the test and development experience. On this basis, the Marquardt engine has been selected as the prime engine, and the RMD C-1 engine was chosen as a close alternate, if required.

In addition to the analysis performed to ensure integrity of the selected engine, Marquardt also indicates that the engine and valves are compatible with program sterilization requirements.

The R-4D rocket engine will provide a 275 sec minus three sigma minimum vacuum specific impulse at 100 lb_f thrust using N₂O₄ and MMH propellants at an oxidizer-to-fuel ratio of 1.6 and a nozzle expansion ratio of 40:1, as required.

Before delivery of the engine to Martin Marietta, the engine contractor will perform engine hot fire characterization by pre-acceptance testing the engine. Marquardt will supply the following data: propellant flow rates, characteristic velocity, thrust coefficient, chamber pressure, valve inlet pressure, thrust, specific impulse, oxidizer-to-fuel ratio, propellant temperatures, test cell pressure, and test profile. The engine will be hot fire characterized using a full bell with area ratio of 40:1. The thrust coefficient obtained will be used to correct for site level firing of the engine following sterilization testing of the pre-packaged system. The engine bell will not be used in the system site level hot fire demonstration test,

A copy of the Components and System Test Plan, MCR-67-20, has been sent to Marquardt for review and approval. Interface requirements have been established and only the detailed configuration of the engine control valves remains as an open item,

The engine will be purged with GN₂ following the sterilization system hot fire demonstration test. Subsequently the engine will undergo Freon MF flush of the oxidizer side and methyl alcohol flush of the fuel side to decontaminate it for handling and inspection.

Table 2 Final Engine Selection

Test Experience	Reliability	Engines Qualification Status	Complexity	12%ETO 38% Freon 12 Compatibility at 50°C (122°F)	Physical Property Heat Cycle Compatibility Extended Duration (@ 135°C (275°F))	Compatibility of Dissimilar Material Thermal Expansion	System Design Suitability	High Temperature Component Exposure	Satisfies Program Performance Requirements
<p>Maxquardt R-4D</p> <p>No. of Altitude Tests: 633 No. of Altitude Starts: 1,141,000 Accumulated Burn Duration: 489,400 sec Total Valve Cycles: 5,812,800</p> <p>NTO/A-50</p> <p>Maximum Duration, One Engine: 13,000 sec No. of Starts: 103,000 Maximum Steady-State Duration: 2100 sec</p> <p>NTO/MMH</p> <p>Three Qualification Engines</p> <p>No. of Altitude Starts: 26,864 Accumulated Burn Duration: 2136 sec</p> <p>Two Development Engines</p> <p>No. of Altitude Starts: 5000 Accumulated Burn Duration: 2000 sec Maximum Steady-State Duration: 1020 sec</p> <p>Rating: 10</p>	<p>Engine</p> <p>NAI: 5518 Cycles 0.935 @ 50% Confidence 0.983 @ 90% Confidence</p> <p>Valve</p> <p>0.996 @ 50% Confidence</p> <p>The reliability is the same for A-50 and MMH since propellant change affects only performance</p>	<p>Developed and qualified for SM, IEM, and Lunar Orbiter with NTO/A-50.</p> <p>Supplemental qualification completed for NTO/MMH for use on SM, 445 engines delivered with 208 remaining to be delivered.</p>	<p>Injector</p> <p>8 valves Individual fuel and oxidizer solenoid valves.</p> <p>Shrink fit injector assembly, small injector</p> <p>Swageplate</p> <p>clogging and distortion. 8 sets, multi-triplet type with one set preignition.</p>	Compatible	Compatible	Compatible	Supply pressure: 180 psi. Existing thrust chamber pressure pickup adequate.	Four solenoid valves; two fuel, two oxidizer subjected to 250°F nonoperating temperature and evaluated. Mode-grade function evidenced.	Satisfactory
<p>RMD C-1</p> <p>NTO/A-50 and NTO/MMH</p> <p>No. of Tests: 4092 No. of Starts: 431,000 Accumulated Burn Duration: 148,000 sec (60% of above tests with NTO/MMH)</p> <p>NTO/MMH</p> <p>Maximum Duration for One Engine: 12,706 sec No. of Starts: 9920 Maximum Steady-State Duration: 1400 sec</p> <p>Rating: 8</p>	<p>Engine</p> <p>Required to demonstrate 69 successful engine firings. Most severe duty cycle: 28 engines/20 environmental engines. Test to failure: 25 of 48 engines total. 0.99 @ 50% Confidence.</p>	<p>Developed and to be qualified by 15 July 1967, as backup for SM, LEM, S-IVB, and AAP. Uses both NTO/A-50 and NTO/MMH.</p> <p>Over 100 engines in program; some already delivered</p>	<p>Minimum</p> <p>Valve</p> <p>Single integrated torque motor operated bipropellant valve.</p> <p>All welded injector assembly moderate injector orifice holes. 4 sets, vortex type.</p>	Compatible	Compatible	Compatible	Supply pressure: 179-191 psi. Existing thrust chamber pressure pickup adequate.	Valve functionally tested with average coil temp at 500°F without detrimental effects.	Satisfactory
Rating: 10	9	7	10	1	10	10	10	9	10 Rating 97
Rating: 8	9	7	10	1	10	10	10	10	10 Rating 94

B. SYSTEM DESIGN AND ANALYSIS

System components have been selected and detailed design of the module and component procurement has been initiated during this quarter. In cases where components changed in size or shape, the system design was revised. A major structural change was caused by a change in the mounting provisions on the propellant tanks. The original intent was to mount the tanks to the rectangular truss in the horizontal plane with the tank inlets and outlets on the vertical axis. The selected tanks have no mounting provisions other than the inlet and outlet bosses. Since the outlets must remain on the vertical axis, truss work was added to carry the mounting loads from the inlet and outlets to the main rectangular truss (Fig. 1).

During this report period, the 600-hr material compatibility tests were completed, allowing final selection of components. In particular, the expulsion devices to be used with each propellant were established and procurement action was started. A Teflon-laminated hemispherical diaphragm will be used in the oxidizer tank and a screen trap assembly will be used in the fuel tank. In addition, aluminum alloy hand valves and ordnance operated valves will be used even though there is a mild incompatibility of aluminum with N_2O_4 at elevated temperatures. The alternative to this is to use titanium components exclusively in the oxidizer system, which is prohibitively expensive for this program.

A schematic that currently represents the system is in Fig. 2.

Several revisions were made to the design criteria to reflect component changes occurring immediately after final component selection. The gas bottle initially selected was later rejected because the material (titanium 7Al-4Mo) was extremely difficult to weld. Two other titanium (6AR-4V) bottles were considered that had different volumes and pressure ratings than the first bottle. This required recomputation of nitrogen loads and system operating pressures. The three bottles are compared in the following tabulation.

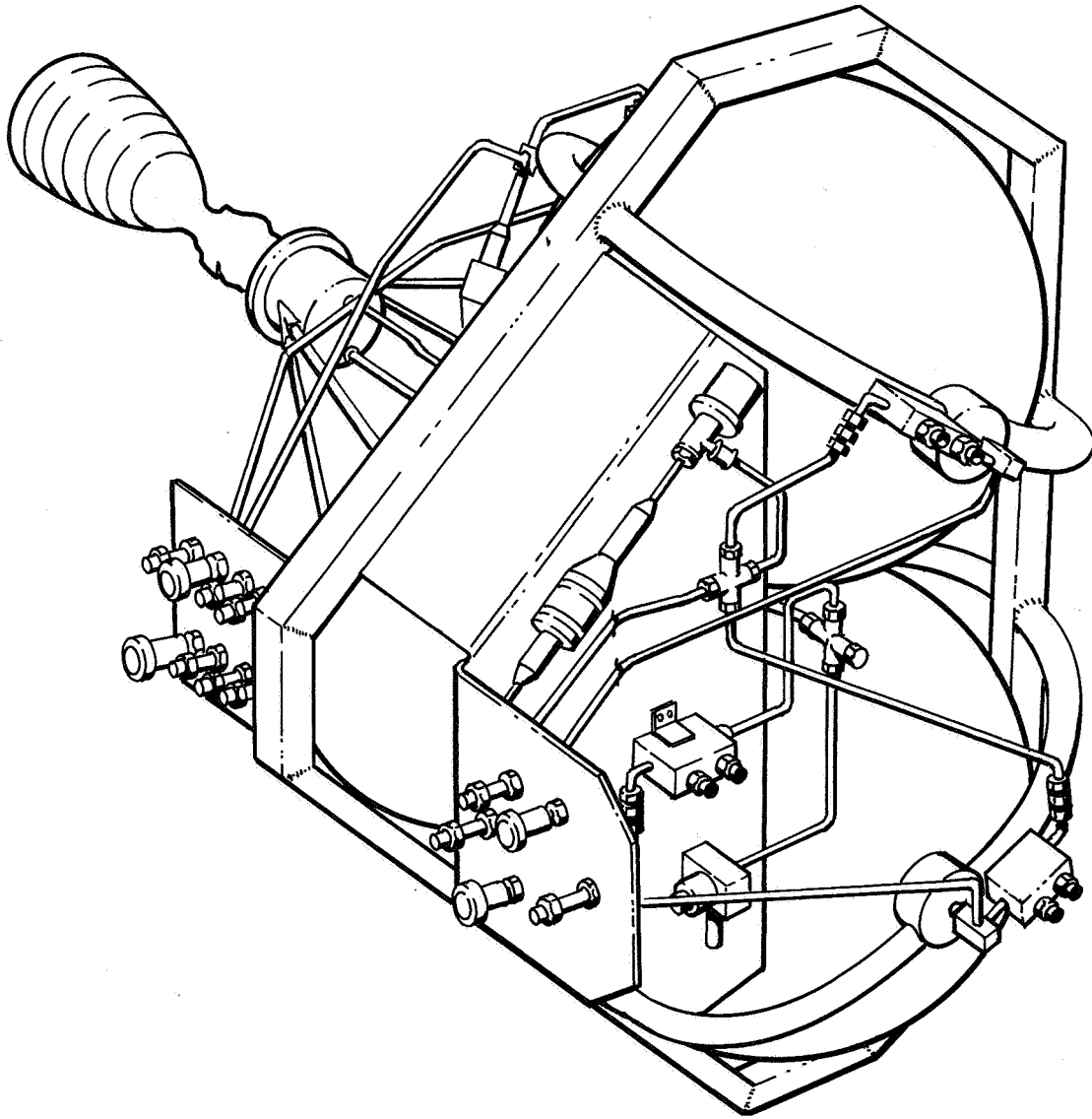


Fig. 1 Artist's Concept of Planar Configuration

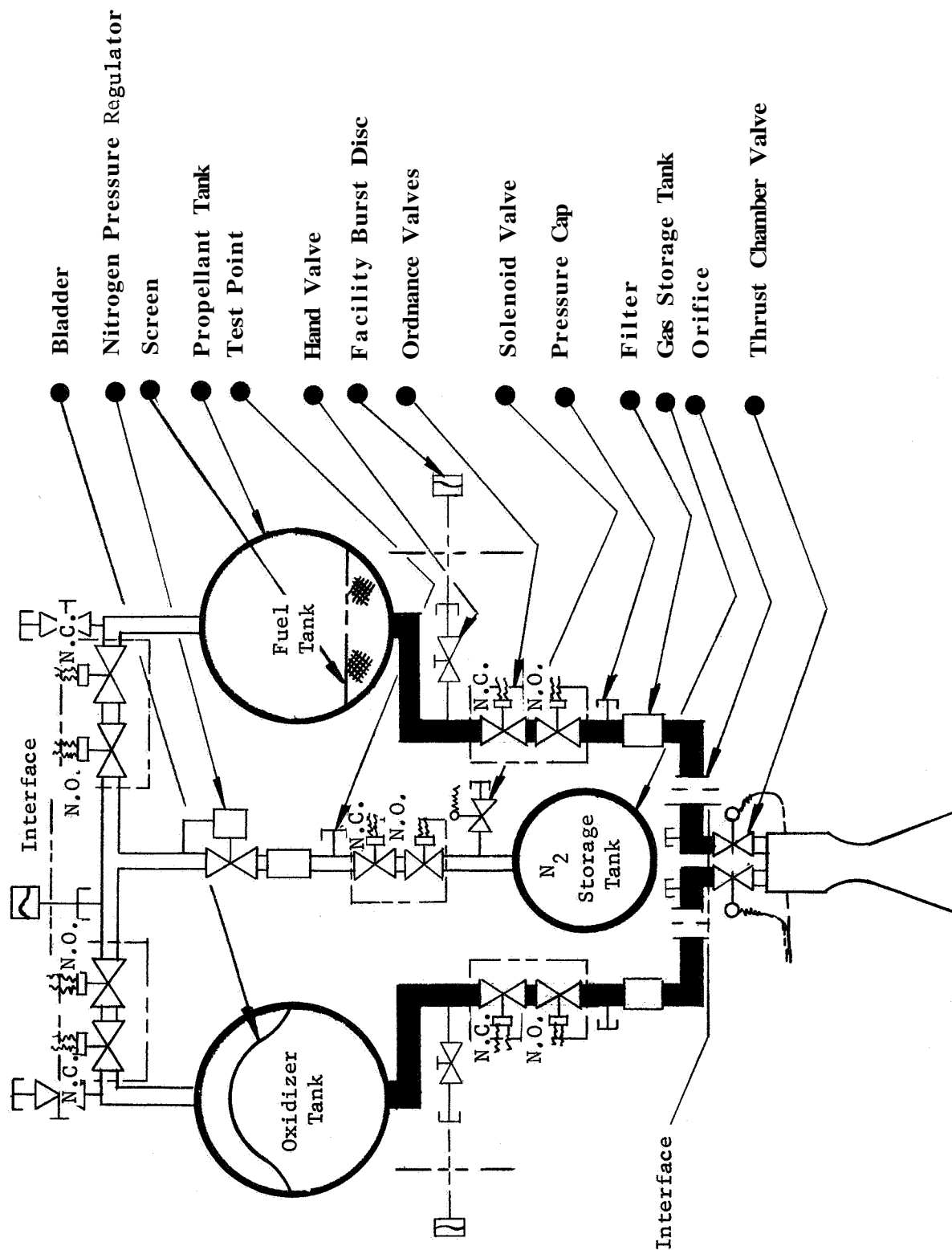


Fig. 2 Sterilizable Liquid Propulsion System

Item of Comparison \ Bottle	First Selection Menasco 785000-503	Pressure Systems 80009-4	Final Selection Menasco 812500-501
Material	Titanium 7Al-4Mo	Titanium 6AQ-4V	Titanium 6Al-4V
Volume (cu in.)	1272	900	1275
Load Pressure (psia)	1475 \pm 25	2150 \pm 50	1650 \pm 50
Proof Pressure (psia)	4500	6700	4875
Mass Loaded (GN ₂) (lb _m)	5.35	5.51	5.45
Mass Required (lb _m)	3.56	3.56	3.56
Margin (lb _m)	1.75	1.95	1.89

During this report period the problem of corrosion of stressed titanium was revealed. The mechanism involves corrosive attack by chlorine in any form that is not highly ionized. Since the Freon 12 component of the decontaminant gas mixture contains chlorine, its use is not recommended with titanium components. An investigation was conducted to find suitable substitutes for the Freon 12. Several commercially available decontaminants were found that were combinations of carbon dioxide and ethylene oxide. Carboxide, a mixture of 90% carbon dioxide and 10% ETO, is non-flammable in air but requires a high chamber pressure (56 psia) to maintain the proper ETO concentration of 600 mg per gaseous liter. Oxyfume 20 and Oxyfume 30 are mixtures of 20 and 30% ETO, respectively, with carbon dioxide and are both flammable in air. Another approach to the problem involved the search for a direct replacement for the Freon 12 in the mixture. Freon C 318 appears to meet all the necessary requirements of not containing any chlorine, not being flammable in air, and requiring a chamber pressure of only 15 psia. A proposal to use this material and a materials compatibility evaluation program has been submitted to JPL for approval.

C. COMPONENT DESIGN AND ANALYSIS

At the beginning of the report period proposals were received from component suppliers. Each design was rated using the system established during the earlier portions of the program. This section presents the ratings and any additional pertinent comments.

1. Pressurant Tank (Table 3)

The Menasco tank, P/N 812500-501, was selected because it scored higher than the other supplier tanks, primarily in the area of test experience. Another Menasco design, P/N 785000-503, had been initially selected because it had better mounting provisions and lower cost. However, the material was titanium, 7Al-4Mo, which is extremely difficult to weld. A decision was made not to risk the welding problems despite the lower cost. Only a single bottle fabricated from the 7Al-4Mo material existed. In addition, Menasco indicated that no additional bottles of this material would be made, therefore, loss of the bottle at any point in the program would require a change to another bottle configuration.

Table 3 Pressurant Gas Sphere Selection

	Airtek	Pressure Systems	Menasco Co
1) Basic Design Analysis			
a) Envelope Size (-15 → +15)	15	15	15
b) Mounting Provisions (-15 → 4-15)	0	0	0
c) Structural Capability (0 → 10)	15	10	7
d) Complexity (0 → 10)	10	10	10
2) Material of Construction (0 → 10)	10	10	10
3) Leakage			
External Leakage (0 → 10)	10	10	10
4) Cycle Life (0 → 15)	0	0	0
5) Vendor			
a) Delivery Target Date (One negative point for each week past target date)	0	0	0
b) Cleaning (0 → 5)	5	5	5
c) Previous Experience at Building Tanks (0 → 10)	10	10	10
6) Weight (0 → 5)	5	5	5
7) Qualification Status			
a) Degree of Testing in Compliance with JPL 30250B (0 → 15)	0	5	10
b) Changes Required (0 → 10)	10	10	10
Total Points	90	90	102
Final Selection: Menasco Company P/N 812500-501			
Cost: \$2380.00			
Modification Required: None.			

2. Propellant Tanks (Table 4)

Final selection of propellant tanks was even more positive than the technical rating score indicates. The tank design selected is the same design as used in the JPL Advanced Lightweight Pressurization System (ALPS) Generant Tank Program with a few minor design changes. The inlet and outlet ports will be strengthened to accommodate mounting provisions. In addition, the forgings that will be used for the fuel tanks will allow extra metal near the outlet port. This extra metal will permit machining of a ring to allow welding of the screen trap to the tank. The bladder material will be Teflon rather than butyl or ethylene propylene compounds as used in the ALPS program.

3. Expulsion Bladder (Table 5)

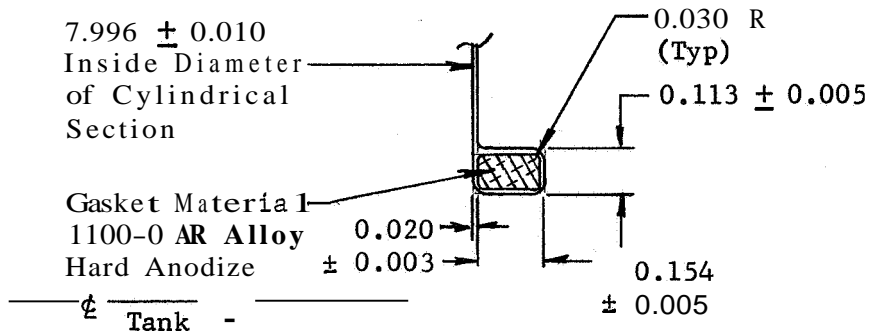
Final selection of a bladder for the oxidizer tanks involved more the selection of a type of bladder than the selection of a supplier. Both metal and Teflon designs were considered. The Teflon design was selected primarily because of its qualification status and cycle life capability. The Teflon bladder (Fig. 3) will consist of laminates of TFE (4 mils thick) and FEP (4 mils thick). It will also incorporate a crown of FEP (0.030 to 0.035 in. thick) at the gas inlet area to prevent extruding of the Teflon through the barrier plate located in the inlet port.

Table 4 Propellant Storage Sphere Selection

	Pressure Systems	Menasco
1) Basic Design Analysis		
a) Envelope Size (-15 → +15)	15	15
b) Mounting Provisions (-15 → +15)	0	0
c) Structural Capability (0 → 10)	10	10
d) Complexity (0 → 10)	10	10
2) Material of Construction (0 → 10)	10	10
3) Leakage		
External Leakage (0 → 10)	10	10
4) Cycle Life (0 → 15)	0	0
5) Vendor		
a) Delivery Target Date (One negative point for each week past target date)	0	0
b) Cleaning (0 → 5)	5	5
c) Previous Experience at Building Tanks (0 → 10)	10	10
6) Weight (0 → 5)	5	5
7) Qualification Status		
a) Degree of Testing in Compliance with JPL 30250B (0 → 15)	0	0
b) Changes Required (0 → 10)	5	0
Total Points	80	75
<p>Final Selection: Pressure System, Inc. P/N 80011-1</p> <p>Cost: \$4,250.00 each plus estimated cost for modification \$500.00 each.</p> <p><u>Modification Required :</u></p> <ol style="list-style-type: none"> 1. The propellant tank out port must be drilled to a large diameter size. 2. Mounting bosses must be provided 3. Change bladder material. 4. Barrier plate at gas inlet, holes drilled smaller. 		

Table 5 Expulsion Bladder Selection

	Ardé Engineering	Dilectrix
1) Basic Design Analysis		
a) Design Concept (-20 → +20)	20	20
b) Mounting Provisions (0 → 10)	10	10
c) Structural Capacity (0 → 10)	10	5
d) Complexity (0 → 10)	0	5
2) Materials of Construction (0 → 10)	0	5
3) Permeation Characteristic		
a) Nitrogen (0 → 2)	2	0
b) Monomethyl Hydrazine (0 → 3)	3	0
4) Performance Cycle Life Capability (0 → 10)	0	10
5) Vendor		
a) Previous Experience at Building Bladders (0 → 10)	10	10
b) Delivery Target Date (One negative point for each week past target date)	0	0
6) Qualification Status		
Previously Qualified Similar Type Hardware (0 → 40)	0	40
Total .Points	55	105
Final Selection: Dilectrix		
Cost: \$3000 each		
Modification: New Design (in size only)		

Detail "A"

Note: All dimensions in inches.

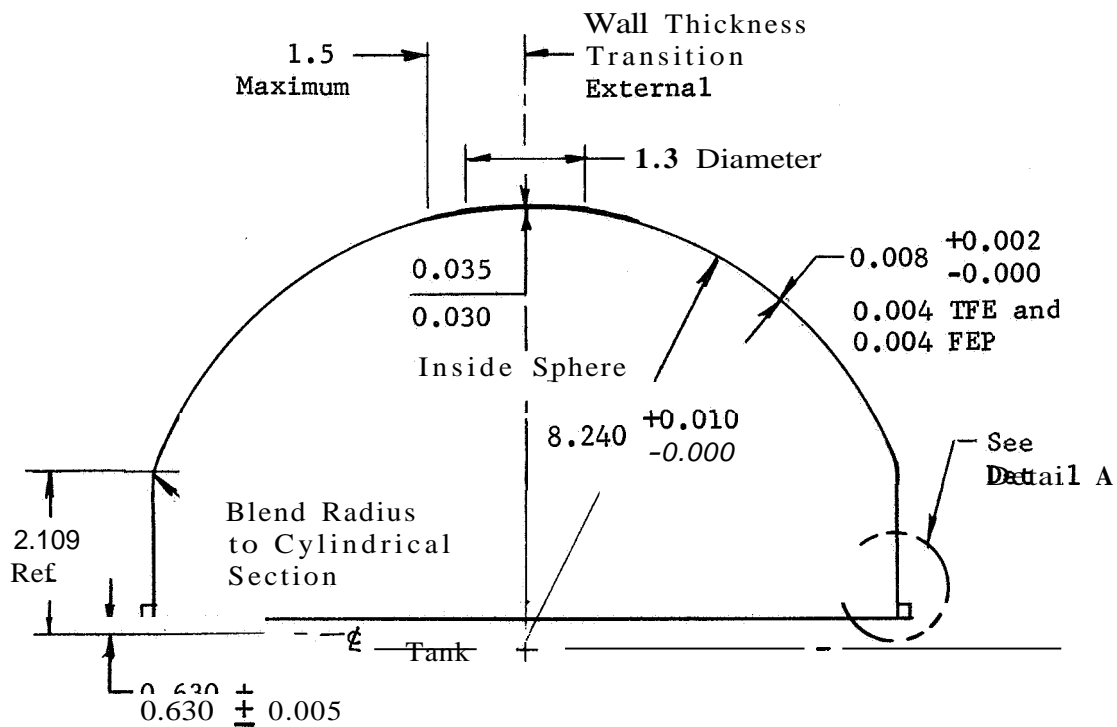


Fig. 3 Teflon Bladder

4. Screen Trap (Fig. 4)

A screen-trap design was established and the requirements were submitted to Western Filter Company for proposal. No other company is known to have experience in the design and manufacture of surface tension devices. Since the trap was designed by Martin Marietta Corporation, consideration is being given to building the trap in-house.

Since the tank material is titanium, an attempt was made to find a supplier of titanium woven screen in the mesh size necessary to support at least 2 in. of MMH. It was found that screen of this size is beyond the current state-of-the-art in both wire drawing and weaving. The material becomes highly susceptible to corrosion in small diameters and is quite brittle, making weaving extremely difficult. Further investigation revealed etched titanium screen was available in proper mesh sizes although the material thickness was a problem. The supplier could etch hole diameters no smaller than the material gage. Samples of material 0.001 in. thick etched to the required mesh size were obtained and exposed to fuel (MMH) at elevated temperature (275°F) with no material degradation or fuel decomposition. Welding of this etched foil into a trap assembly, however, was a potential problem requiring a welding development program. One alternative solution was available: use titanium sheet to build up a frame assembly and attach stainless steel screen window assemblies using a crimping, riveting, or bolting technique. A seam welding technique was developed in-house to form a joint, as shown in Fig. 4. The stainless steel screen is sandwiched between sheets of titanium. A seam weld is made outside the screen to fuse titanium to titanium and a second seam is made through the screen. This latter weld does not provide complete fusion of the two metals; however, it does provide a good mechanical bond and seals the joint against fuel leakage around the edge of the screen. Fuel at elevated temperature testing will be done with this joint during the next period, and if no decomposition problems are evident screen traps of this configuration will be made. The screen traps will be conical in shape with an entrapped volume of approximately 54 cu in. They will consist of a flat 9.4-in.-diameter circular titanium sheet with a 2-in.-square port cut in the center, jointed at its edge with a 8.5-in. conical dish-shape titanium sheet. Four rectangular ports will be cut into the dish segment with stainless steel screening welded over the ports using the described welding technique.

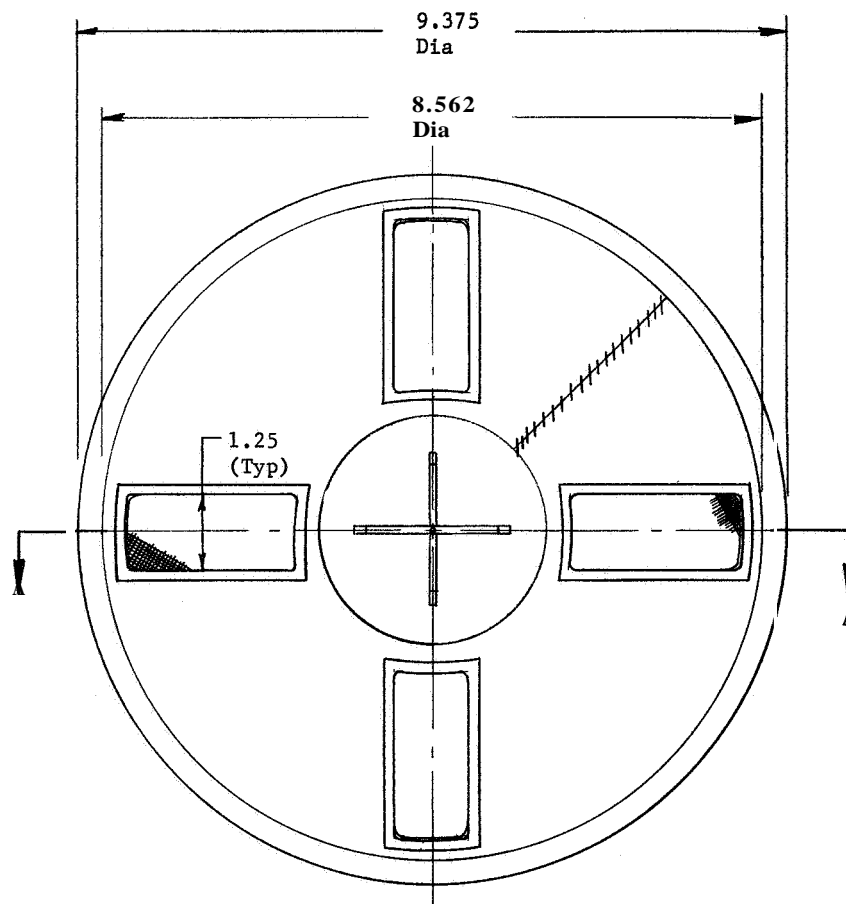
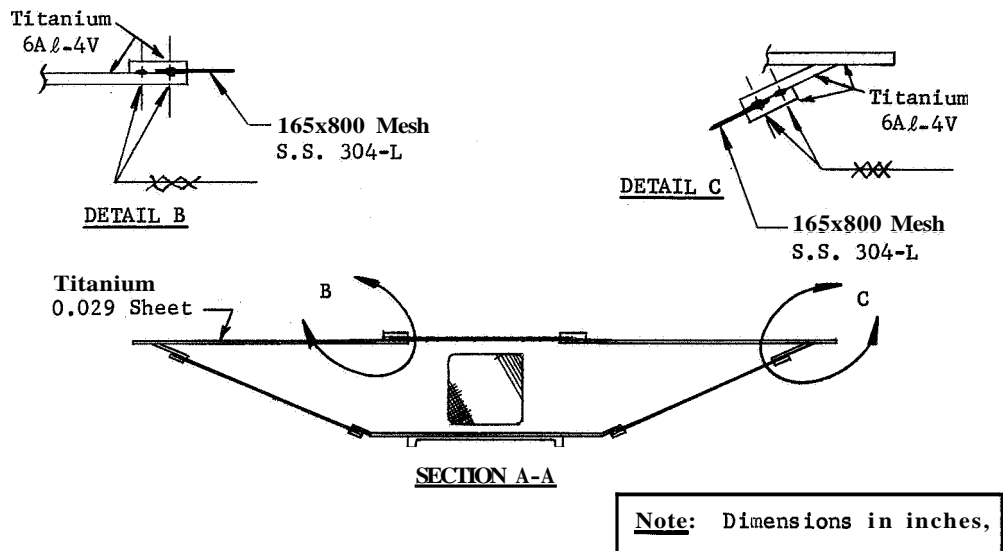


Fig. 4 Screen Trap

5. Hand Shutoff Valve (Table 6)

Results of the 600-hr compatibility program indicated that iron or nickel bearing alloys could not be used in contact with oxidizer. In addition, aluminum was mildly incompatible with N_2O_4 at 600 hr, but not at 300-hr exposure. Only titanium proved to be a completely compatible metal. All proposals originally received indicated use of steel hand valves. A second round of proposal requests indicated no titanium component designs and only a single aluminum design. Therefore, despite the partial incompatibility, the aluminum valve design by Vacco was selected. This decision was influenced by the fact that although corrosive action would occur on the aluminum the propellant would not be degraded as would be the case with steels. Since both the customer-supplied aluminum ordnance valves and the hand valves were over-designed structurally, the attack could not cause structural failure. If a flight system were to be built, titanium components should be used throughout that part of the system exposed to oxidizer (N_2O_4) during heat sterilization.

6. Gas Pressure Regulator (Table 7)

The selected design, Sterer Dwg 35570, was chosen in preference to Sterer Dwg 23010 because it is a proven and qualified design. It is basically the same design as that used on Mariner II (Sterer Dwg 18910). Material in the cap and ball reseating pin was changed from 2024 T4 AR to stainless steel. Other changes include the addition of a 10μ filter on the inlet side, change of inlet and outlet ports, and change of pressure setting to conform to the present application.

7. Solenoid Valve (Table 8)

The chosen design was selected because it is basically the same as Sterer Dwg No. 31580, which was qualified for use on the biosatellite. Minor changes include the substitution of Kynar for a nylon threadlock. This threadlock is in a noncritical area and is backed up by a final wire lock. The solenoid potting compound was changed to one that is compatible with ethylene oxide and the ports were changed to conform to the present application,

This valve is a good design for a fill-and-drain valve in that both the inlet and outlet ports are protected by filters, thereby reducing the possibility of seat contamination during both the fill and drain operation. Also, since this valve must be closed during the thermal cycling, the all-metal seat precludes any degradation by cold flow that might be present in a soft seat design.

Table 6 Hand Shutoff Valve Selection

	NVB32181 Vacco Valve
1) Basic Design Analysis	
a) Insensitivity to Thermal Change (-5 → +5)	+3
b) Stem Seal Design (-10 → +20)	0
c) Seat Seal Design (0 → 5)	3
d) Structural Capability (0 → 10)	Unk.
e) Complexity (0 → 10)	6
2) Materials of Construction (0 → 10)	10
3) Leakage	
a) Internal (0 → 5)	4
b) External (0 → 5)	5
4) Performance	
a) Flow (0 → 5)	3
b) Pressure Drop (0 → 5)	3
5) Vendor	
a) Previous Experience Required Minimum Development (0 → 10)	5
b) Delivery (one negative point for each week past target date)	0
6) Envelope and Weight (0 → 5)	4
7) Qualification Status	
a) Degree of Test in Compliance with JPL 30250 (0 → 20)	0
b) Changes Required (0 → 20)	15
Totals	61
Final Selection: Vacco Valve Company P/N NVB32181	
cost: \$220.22 each	
Modifications Required: None	

Table 7 Gas Pressure Regulator Selection

	Sterer Dwg 23010	Stratos Dwg 337001	Calmec Dwg 820	Sterer Dwg 35570	Natio l Water ift	Royal Ind Dwg 12077
1) Basic Analysis						
a) Insensitivity to Thermal Changes (-10 → +10)	10	10	10	10	8	10
b) Protection of Small Orifices (-10 → +10)	10	0	5	5	8	-10
c) Complexity (0 → 5)	4	4	5	5	0	5
d) Seat Design (0 → 5)	5	2	2	5	5	2
e) Structural Capability (0 → 10)	10	8	10	10	10	10
2) Materials of Construction - Compatibility (0 → 10)	10	10	10	10	10	10
3) Leakage						
a) Internal (0 → 5)	5	3	2	4	4	2
b) External (0 → 5)	5	3	5	4	5	5
4) Performance						
a) Regulation Pressure Band/Width (0 → 10)	10	5	8	10	5	8
b) Overshoot on Lockup (0 → 5)	5	3	4	4	4	4
c) Overshoot on Inlet "Squib Valve" Initiation (0 → 5)	5	4	3	3	4	3
d) Pressure Band Drift Due to Environmental Changes (0 → 5)	5	5	5	5	5	5
e) Allowable Inlet Pressure Variation (0 → 10)	10	10	10	10	8	10
5) Vendor						
a) Previous Experience Requiring Minimum Development (0 → 10)	10	5	5	10	10	5
b) Delivery (one negative point for each week past target date)						
6) Envelope and Weight (0 → 5)	5	5	5	5	5	5
7) Qualification Status						
a) Degree of Testing in Compliance with JPL 30250B (0 → 20)	8	5	5	10	10	5
b) Changes Required (0 → 20)	18	10	18	1%	10	10
Total	135	92	112	12%	111	89

Final Selection: Sterer Dwg No, 35570

Cost: \$3,455 each.

Table 8 1/4-in. Solenoid Valve Selection

[illegible]

8. Filter (Table 9)

The Vacco filter and Western filter designs were rated equal on a technical basis as shown in Table 9. However, the Vacco filter cost more than double the Western filter and was thus down-rated to second choice. All filter designs designate stainless steel, but this is not a problem since the filter assemblies will be isolated from propellants during the heat sterilization cycles.

Table 9 Filter Selection

	APM (AC-H1QZ-810X1)	Acoustica (34007)	Vacco (S3-81840)	Western (20477-10)	Fluid Dynamics FK2968-10
1) Basic Analysis					
a) Insensitivity to Thermal Change * (-10 → +10)	+5	0	+5	+5	+5
b) Filter Element Design and Installation (0 → 10)	8	2	8	8	7
c) Assembly Sealing Design (0 → 10)	8	2	8	8	8
d) Structural Capability (0 → 10)	5	5	5	5	5
e) Complexity (0 → 10)	7	4	10	8	8
2) Materials of Construction (0 → 10)	7	5	7	7	7
3) Leakage					
a) Internal (0 → 5)	5	5	5	5	5
b) External (0 → 5)	4	2	4	5	4
4) Performance					
a) Flow Rate (0 → 5)	4	4	4	4	4
b) Pressure Drop (-5 → +5)	3	3	2	3	3
c) Effective Area (0 → 5)	3	4	3	3	2
5) Vendor					
a) Previous Experience Requires Minimum Development (0 → 10)	10	5	8	8	8
b) Delivery (one negative point for each week past target date)	0	0	0	0	0
6) Envelope and Size (0 → 5)	3	0	4	4	5
7) Qualification Status					
a) Degree of Test in Compliance with JPL 30250 (0 → 20)	0	0	0	0	0
b) Changes Required (0 → 20)	10	5	20	20	18
Totals	82	46	93	93	89
Final Selection: Western Filter Co., Inc. P/N 20477-10					
Cost: \$237.00 each					
Modifications Required: None					

D. MATERIAL INVESTIGATIONS

Materials investigation has continued throughout the period. The 600-hr material screening tests were completed and are reported upon in the Appendix of this report. In addition many prescreening and special test results are presented.

During the next period the long-term storage tests will be started and will continue throughout the remainder of the program. Sufficient testing during the material screening program has been completed to allow final selection and procurement of all system components with the exception of the screen trap. Testing of the stainless steel screen-to-titanium sheet joint will be completed during the next report period,

E. TEST PLAN

During the period the test plan for the component and system test phases of the program was completed. This document, MCR-67-20, was issued in final form and submitted to JPL for approval. It defines test equipment and test sequences and provides flow charts and a test schedule for each testing phase. The plan will be modified, if required, as detailed planning and test fixture design proceed.

F. TEST FACILITIES

1. Decontamination/Sterilization Chamber

Mechanical design of the chamber and its subsystems was completed. A design review of the system was conducted, and all drawings have been released to begin fabrication. Before the chamber design was made final, an analysis of chamber performance with a 12% ETO/88% Freon C318 sterilant gas was completed. The existing chamber will perform satisfactorily on the alternative sterilant gas, if that gas is used.

Design of the combined decontamination and sterilization chamber has been modified so that the electrical heaters (heat source for sterilization tests) will be removed from the chamber

during ETO decontamination tests. A tradeoff study was made between the increased costs of providing ETO-compatible electrical heaters, cabling and pressure-tight pass-throughs versus the cost of using noncompatible, removable heaters at the expense of having to run two chamber qualification tests. The study favored the two-geometry system, and the existing chamber design accordingly features a removable electrical heater array. The heater array will be removed when the chamber is used for ETO decontamination. The chamber will be qualification-tested for both geometries,

Fabrication of hardware for the decontamination/sterilization chamber and the associated heating, cooling, humidifying, and ETO injection systems is in progress.

The control system design for the chamber is being finalized. In addition to the primary function of controlling the chamber environment to the conditions required for decontamination and heat sterilization, the control system is being designed to effect a safe, automatic shutdown of the chamber if any of the following conditions occur:

- 1) Oxidizer vapor in chamber;
- 2) Fuel vapor in chamber (sterilization only);
- 3) Chamber overtemperature (chamber reference temperature measurement);
- 4) Hot water supply over 56°C (decontamination only);
- 5) Excessive pressure in propellant tanks;
- 6) Power failure.

If a power failure occurs, the chamber system will operate from an independent auxiliary power supply.

Parts fabrication and assembly of the decontamination and sterilization chamber will be in progress throughout April, in preparation for shakedown tests early in May. Chamber qualification tests will follow the shakedown tests.

2. Propellant Vapor Detectors

Compatibility testing of the vapor detectors with the ETO sterilant gas mixture has been completed. The oxidizer vapor detector performed satisfactorily throughout long-term exposures

to the sterilant gas, giving the expected output signal response to periodic injection of 5 ppm N_2O_4 vapor samples. The oxidizer vapor detection system has therefore been incorporated into the sterilization chamber system to initiate the shutdown sequence on detection of N_2O_4 vapor during the ETO decontamination and heat sterilization tests.

Extensive testing of the fuel vapor detector has shown that the device is unsuitable for use with ETO. Initial tests indicated that the detector would start to show anomalous behavior after 3 to 5 hr of exposure to the sterilant gas mixture. An analysis of the chemistry of the detector cell indicated that continuous replenishment of the reagent would maintain the chemical balance (pH) of the cell. A test run made with continuous replenishment of the reagent demonstrated that the chemical balance was in fact maintained; however, the detector exhibited a different kind of aberration that could only be attributed to ETO reaction with the cell electrode. At this point it was recognized that a complete redesign of the fuel detector would be required to make it perform satisfactorily. Such a major effort could not be accommodated within the scope of this program. As a result, a decision was made to eliminate the requirement for fuel vapor detection during ETO decontamination. This decision was based on the following items:

- 1) During ETO decontamination the portion of the module containing fuel is operating with an extremely high factor of safety. This system is structurally identical to the oxidizer system that was designed to operate at 1000 psia. Fuel pressure during decontamination is on the order of 3 psia;
- 2) The existing fuel detector proved unsuitable for use with ETO;
- 3) No alternative system is available;
- 4) Design and development of a suitable system **cannot** be accommodated in the scope of the existing program,

To compensate for the inability to detect a fuel leak in the chamber, a fuel tank overpressure switch has been incorporated in the sterilization/decontamination chamber control system, since the probability of fuel leak could be expected to increase at elevated pressures. This system will activate the chamber shutdown.

3. Humidity Indicator Tests

Tests of the electrohygrometer humidity monitor are in progress. Earlier tests in the sterilant gas at ambient temperature indicated that the system would perform satisfactorily. The test fixture is now being modified to permit testing against the Alnor dew-pointer at 50°C without incurring saturation in the samples drawn into the Alnor.

The electrohygrometer is intended to monitor chamber humidity rather than control the humidity system. The time constant of the water vapor supply system is expected to be such that manual control of the water vapor injection system is mandatory. If the currently planned tests show that the electrohygrometer will not operate satisfactorily in ETO, the Alnor dew-pointer will be used to check relative humidity in the decontamination chamber on a periodic basis.

4. Safety Equipment Compatibility

Testing of personnel safety equipment for short-term compatibility with vapor-phase sterilant gas was completed. The items tested included all parts of a splash suit ensemble and gas masks with two types of canisters. The test results indicated that the existing splash suit ensemble is suitable for personnel protection against sterilant gas spills, and that the existing gas masks and organic vapor removal canisters are suitable for use in an ETO-contaminated atmosphere.

5. Functional Test Fixtures

Design of the test fixtures for the component functional tests in Task II is in progress. All procurement items related to these fixtures have been ordered. Design effort was 80% complete at the close of this reporting period and will be completed in the next period to provide a firm basis for timely completion of the functional test procedures. Parts fabrication and fixture assembly is scheduled for a later point in the program schedule, consistent with planned receipt dates for the components.

6. Test Procedures

Preparation of test procedures for the decontamination, sterilization and chamber qualification tests was in progress at the end of the reporting period. The procedures are being modified concurrent with the finalizing of the control system electrical design.

Procedure preparation will continue through the next reporting period, in readiness for the chamber shakedown tests. Test procedures will also be written for the component functional tests and for the necessary presterilization preparation of components and installation in the chamber.

7. Propulsion Module Thrust Measurement

Originally the thrust measurement was to be made using load cells located at the module-to-test stand interface points. This would require a fairly complex computation of thrust since center of gravity and propellant mass variations would be superimposed on thrust. Several alternative methods were studied to see if a more accurate system could be devised. The system finally decided on consists of washer-type load cells mounted at the engine interface. The engine is supported on the module main rectangular truss by three support rods. The rods attach to a mounting ring bolted directly to the engine. Washers will be mounted between the support rods and the engine attachment ring. This will allow direct measurement of engine thrust since the only loads applied to the washers will be thrust, engine weight, and mounting bolt tension loads. The engine weight and bolt tension loads can be eliminated during cell calibration.

MCR-67-15 (**Issue 2**)

APPENDIX

MATERIALS COMPATIBILITY STUDY

I. INTRODUCTION

A test program was initiated in November 1966 to select materials of construction for a liquid bipropellant propulsion module that could withstand decontamination and dry-heat terminal sterilization environments.

Testing required for selection of materials of construction of the module has been completed. This appendix serves as a final report on materials testing with the exception of long-term storage tests and certain minor confirmation testing.

II. OBJECTIVE

The primary objective of this program was to determine materials suitable for construction of a propulsion module that would be loaded with propellants then subjected to the decontamination and dry heat terminal sterilization cycles described below. A piece parts test program was conducted to provide required data.

Decontamination - Subject hardware to six cycles of 29-hr exposure to the following conditions:

- 1) 12% ethylene/88% Freon 12 environment;
- 2) 35 to 55% relative humidity;
- 3) $122^{\circ}\text{F} \pm 2^{\circ}\text{F}$.

Dry-Heat Cycle - Subject hardware to six cycles of 76 hr of dry heat at 275°F .

To simplify the materials selection test program certain modifications were made to the cycles defined above. These were as follows:

- 1) Decontamination cycle testing was not performed. Sufficient information was available from industry literature to provide compatibility data in this area;
- 2) Dry heat exposure cycles were modified to cause exposure of materials at 275°F for 600 hr continuously with no cooldown. The principal reason for this change was to preclude refluxing action from occurring in the propellant test chamber.

Additional studies were performed, in conjunction with the overall test program, related to safety and to generate test procedures for the completed module. These included:

- 1) Reactivity of the ethylene oxide/Freon 12 atmosphere with propellant vapors in the event of a leak;
- 2) Effect of the ethylene oxide gas on vapor sensors planned for use during the decontamination cycles;
- 3) Passivating procedures for fuel systems to ensure that decomposition of the propellant will not occur as a result of a reactive oxide or a catalytic contaminant;
- 4) Evaluation of propellants after exposure to a material that sustained no attack to verify that the propellant was mutually compatible with the material in the sense that the propellant did not degrade;
- 5) Evaluating effects of Freon 12 on titanium structures.

111. RECOMMENDATIONS

The Marquardt Model R-4D engine should be selected since all of its materials of construction will withstand both decontamination and terminal sterilization cycles.

Propulsion system hardware that will contact propellants during sterilization cycles should be constructed of the following materials:

- 1) Propellant tanks -
 - a) N_2O_4 - titanium (6Al-4V),
 - b) MMH - titanium (6Al-4V), stainless steel (17-4) aluminum (2014-T6 or 2219-T8);
- 2) Plumbing lines -
 - a) N_2O_4 - titanium (6Al-4V),

- b) MMH - stainless steel (304, 304L, 321, or 347), aluminum (2014, 2219, 2024, or 6061);
- 3) Valve bodies -
 - a) N_2O_4 - titanium (6AR-4V) or aluminum with sulfuric or chromic acid anodize on all surfaces that will contact propellant during sterilization,
 - b) MMH - aluminum (2014, 2024, or 6061) or stainless steel (304, 304L, 321, or 347);
- 4) Positive displacement devices for propellants -
 - a) N_2O_4 - Teflon bladder (TFE-FEP laminate) or titanium sieves,
 - b) MMH - stainless steel screen (304 or equivalent) aluminum screen (6061 or 5056) or Teflon bladder (TFE-FEP laminate) ;
- 5) Burst discs
 - a) N_2O_4 - titanium (6AL-4V),
 - b) MMH - aluminum (1100 or 6061), or stainless steel (304, 321, or 347).

Propulsion system hardware that will not be exposed to propellants during decontamination and sterilization cycles should be constructed of the materials listed in the literature survey (contained in the first Quarterly Report) as applicable to the particular design application.

The ASI vapor detector for N_2O_4 is suitable for use in an ethylene oxide atmosphere. The MMH sensor will not function properly and should not be used.

An automatic vent and purge system is required for use during decontamination since either propellant can react with the ethylene oxide. In the event of a propellant leak, a serious pressure rise could occur inside the chamber causing rupture.

The entire MMH system requires stringent cleaning followed by passivation with propellant-water mixture to ensure that reaction of propellants with contamination does not occur after propellant loading. This is especially true for screens and for welded joints that may entrap oxides or other reactive contaminants.

Additional testing of effects of Freon 12 on titanium structures should be conducted. Information available to date is not conclusive.

IV. RESULTS

Chapter V, Procedure, outlines the variety of tests conducted. Results of these tests are presented in this chapter. The data are organized by material and design application, rather than the type or duration of testing performed. No data are listed regarding compatibility of metals in the dry heat cycle unless they were in contact with propellants, since all metals considered were capable of withstanding the temperature.

A. TITANIUM ALLOY

Titanium alloys tested included commercially pure 6AR-4V.

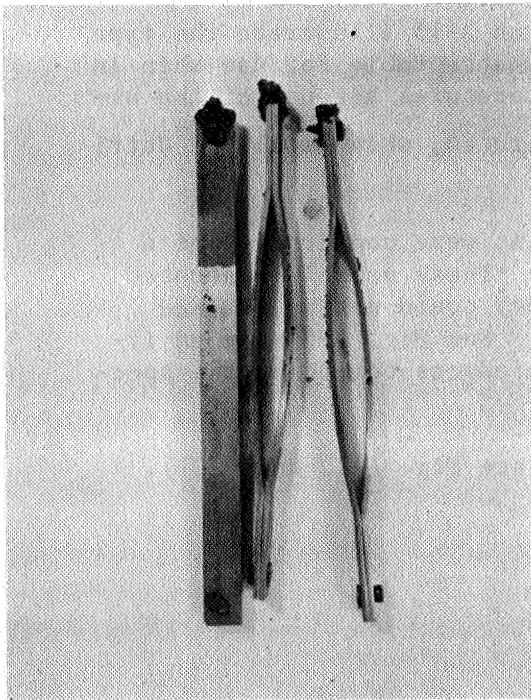
N₂O₄ Exposure - No attack. Figures A-1 and A-15 illustrate the resistance of this material to corrosion.

MMH Exposure - No attack occurred (see Fig. 21).

B. ALUMINUM ALLOYS

Aluminum alloys tested included 1100-0, 2014-T6, 2219-T-87, and 6061-T6.

N₂O₄ Exposure - All alloys were attacked by the propellant resulting (usually) in intergranular corrosion or in pitting. In all instances, a residual corrosion product was formed. This product varied from a white, granular deposit to a thick, viscous, semi-fluid. The products were amorphous.



(a) As Removed from Test



(b) Specimen Section (200X)

Note:

1. Specimens shown in (a) above were prestressed to 50% and 75% of their yield strength.
2. Contamination on specimens is corrosion products from the Monel rivet used to join the ends. No corrosion products of titanium were present.
3. **Section shown in (b) above exhibits no evidence of surface attack, intergranular corrosion, or stress corrosion.**

Fig. A-1 Titanium (6Al-4V) after Exposure to N_2O_4 for 600 hr

Figures A-2, A-3, A-4, A-5, and A-15 illustrate the degree and type of attack sustained. All alloys tested were unacceptable for use when in contact with N_2O_4 at sterilization temperatures except for limited applications discussed in Chapter 111, Recommendations.

MMH Exposure - No attack occurred on any alloy tested (see Fig. A-19).

C. STAINLESS STEEL ALLOYS

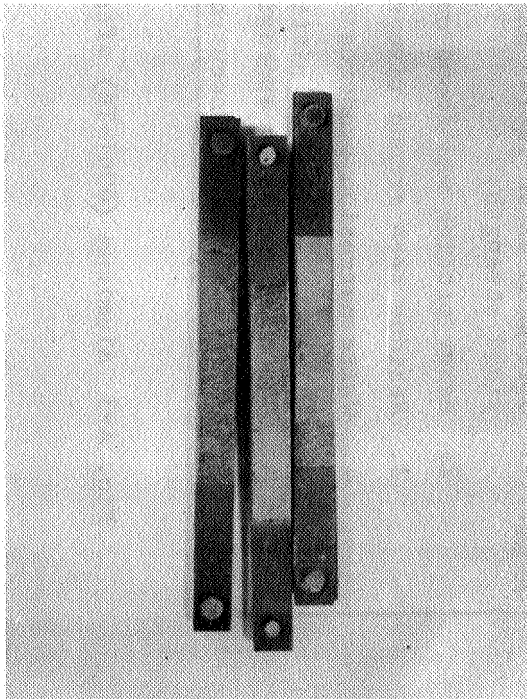
Stainless steel alloys tested included 304, 321, 347, 17-4 PH, and 17-7 PH.

N_2O_4 Exposure - All alloys were attacked by the propellant, resulting in intergranular corrosion and pitting. In all instances, a residual corrosion product was formed. The product was extremely viscous, amorphous on drying, and accelerated corrosion of dissimilar metals, except titanium. Spectrographic analysis of a typical corrosion product indicated that elements present were the same as those contained in the alloy.

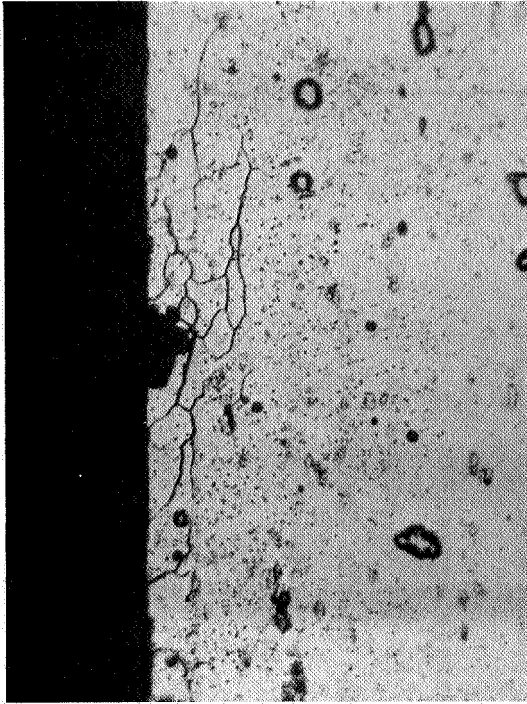
Figures A-6, A-7, A-8, A-9, A-10, and A-15 illustrate the type of attack sustained. All alloys were unacceptable for use when in contact with N_2O_4 at sterilization temperatures because of the attack, as well as the contamination generated, which would be detrimental to the propulsion system.

Test vessels containing the specimens were constructed of a variety of 300 series stainless steels. These vessels generated the viscous deposit to a degree that they could not be drained of propellant through a 1/4-in. outlet. See Fig. A-24 for an illustration of the amount of corrosion products formed on the specimen container rack.

MMH Exposure - No attack occurred (see Fig. A-20 and A-21).



(a) As Removed from Test

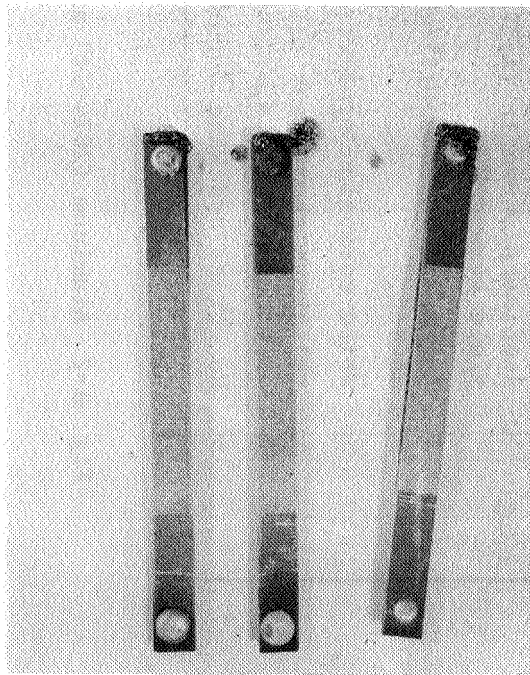


(b) Specimen Section (200X)

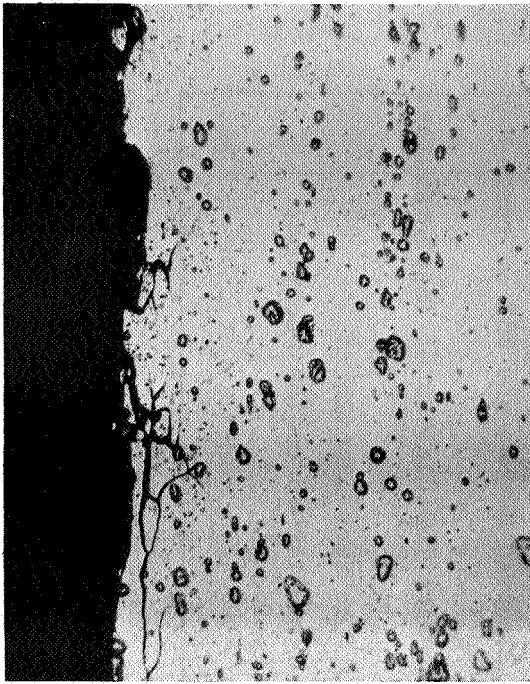
Note:

1. Specimens shown in (a) above were prestressed to 50% and 75% of their yield strength.
2. A general surface pitting was observed with indications of intergranular attack.
3. White corrosion products were found in the test container.
4. (b) above shows that pitting and intergranular corrosion occurred. Depth of attack is approximately 0.0035 in.
5. No stress cracking occurred.

Fig. A-2 Aluminum (2014-T6) after Exposure to N_2O_4 for 600 hr at 275°F



(a) As Removed from Test

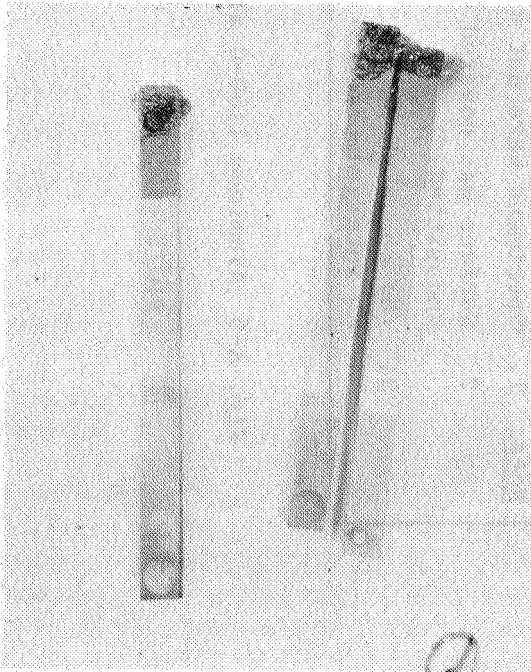


(b) Specimen Section (200X)

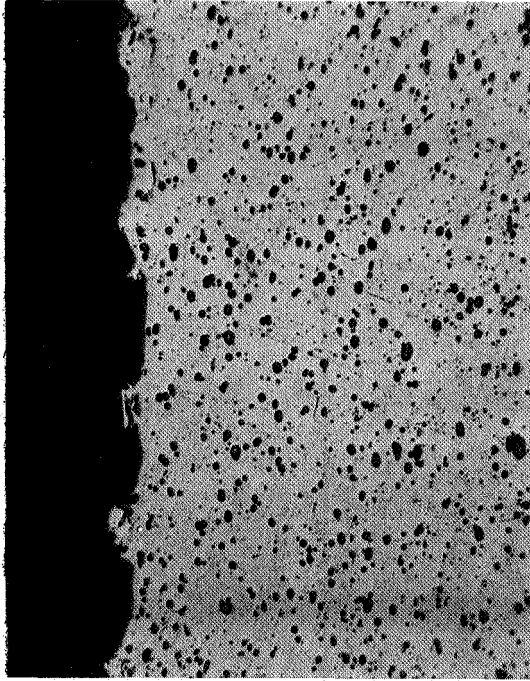
Note:

1. Specimens shown in (a) above were prestressed to 50% and 75% of their yield strength.
2. Heavy pitting was found on all surface areas.
3. Residue seen around rivets is the result of attack on the Monel rivet.
4. White corrosion products were found in the test container.
5. (b) above shows that pitting and intergranular corrosion occurred. Depth of attack is approximately 0.003 in.
6. No stress cracking occurred.

Fig. A-3 Aluminum (6061-T6) after Exposure to N_2O_4 for 600 hr at 275°F



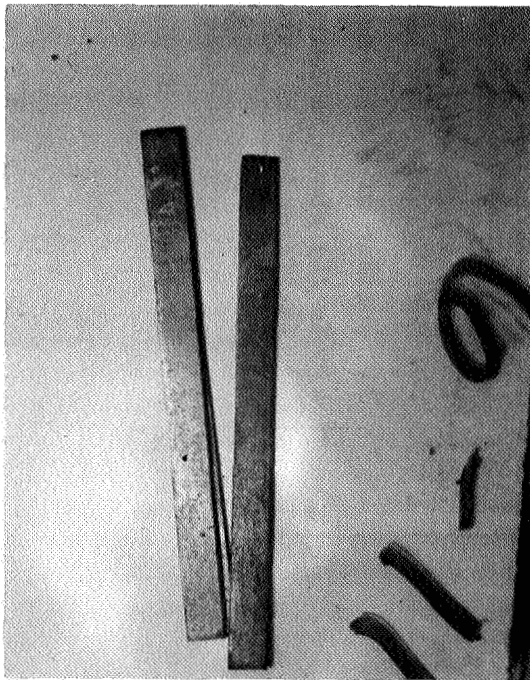
(a) Removed from Test



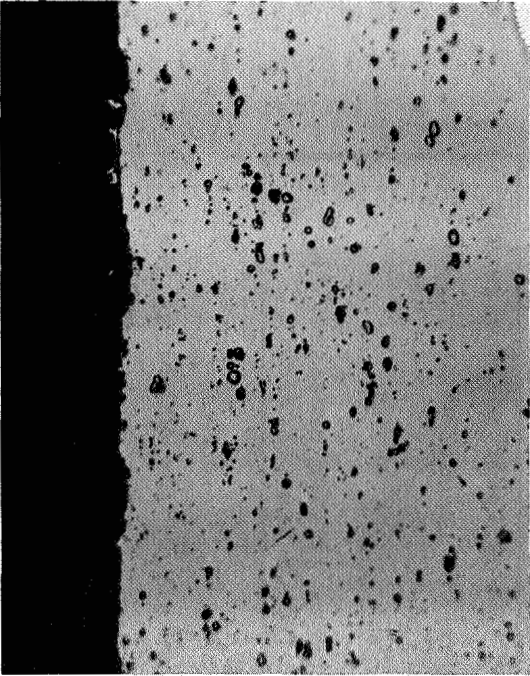
(b) Specimen Section (200X)

- Note:**
1. Specimens were stressed to 50% and 75% of their yield strength.
 2. Deposit around rivets (a) above were the result of attack on the Monel rivets used to fasten the strips.
 3. Surface of specimens were lightly etched.
 4. In (b) above, surface attack and heavy pitting occurred. No intergranular or stress corrosion attack was observed.

Fig. A-4 Aluminum (2219-T87) after Exposure to N_2O_4 for 600 hr at 275°F



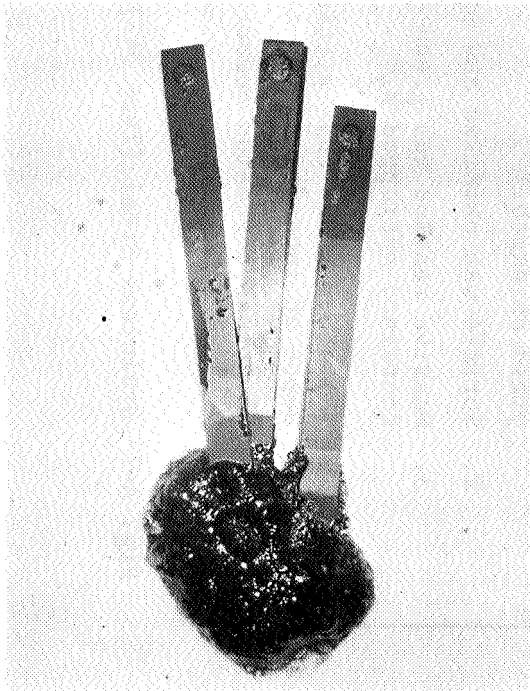
(a) As Removed from Test



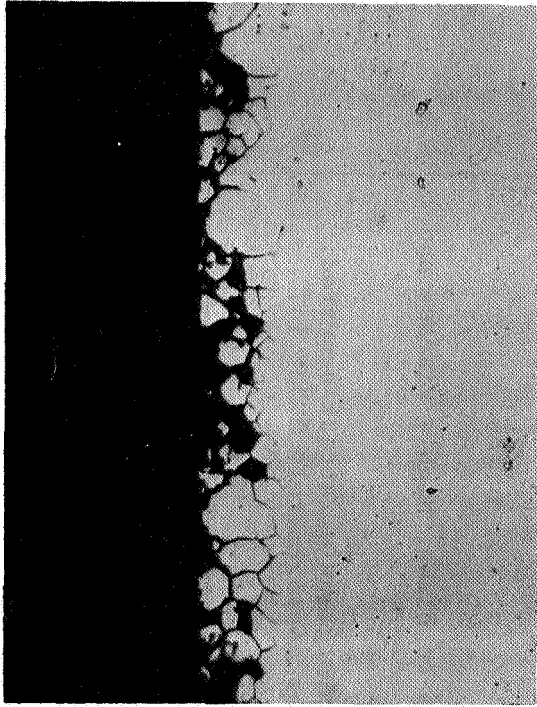
(b) Specimen Section (200X)

- Note:**
1. Specimens were not stressed because of their low yield strength.
 2. Surface was covered with white corrosion products, was oxidized and pitted.
 3. (b) above shows that surface attack resulted in moderate pitting. No intergranular or stress corrosion was noted.

Fig. A-5 Aluminum 1100-0 after Exposure to N_2O_4 for 600 hr at 275°F



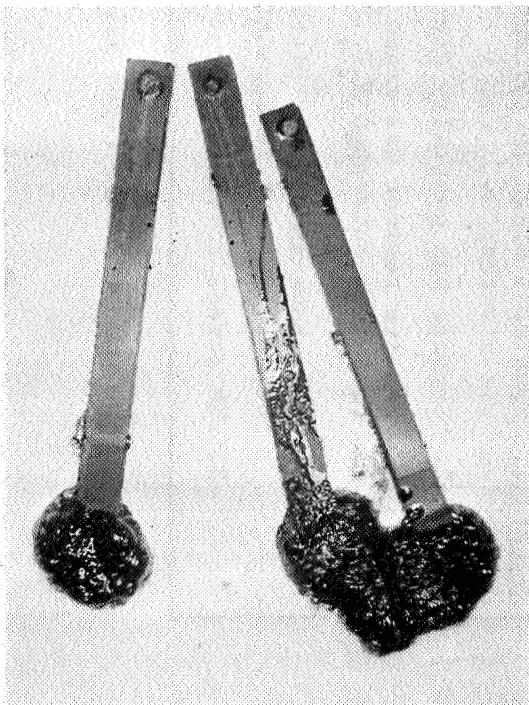
(a) As Removed from Test



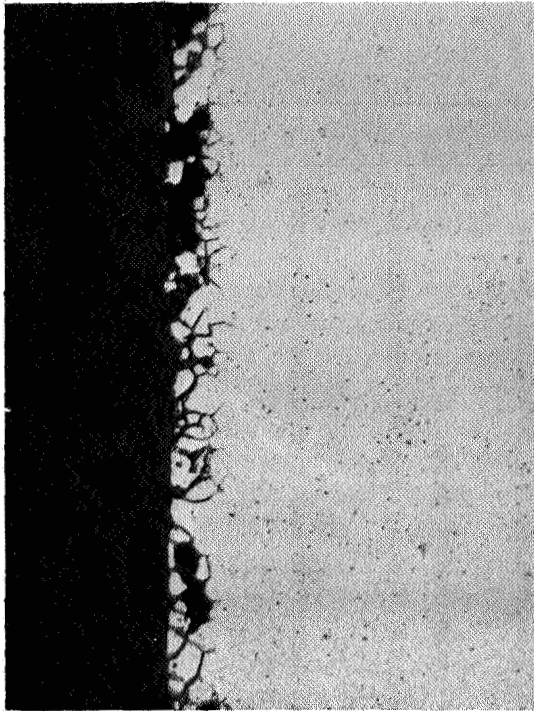
(b) Specimen Section (200X)

- Note:**
1. Specimens shown in (a) above were prestressed to 50% and 75% of their yield strength.
 2. Surface was generally etched with some heavy pits.
 3. Massive residue was green and brown to black, highly viscous and contained entrapped N_2O_4 .
 4. (b) above shows that pitting and intergranular corrosion occurred. Depth of attack is approximately 0.002 in.
 5. No stress corrosion occurred.

Fig. A-6 Stainless Steel (Type 321) after Exposure to N_2O_4 for 600 hr at 275°F



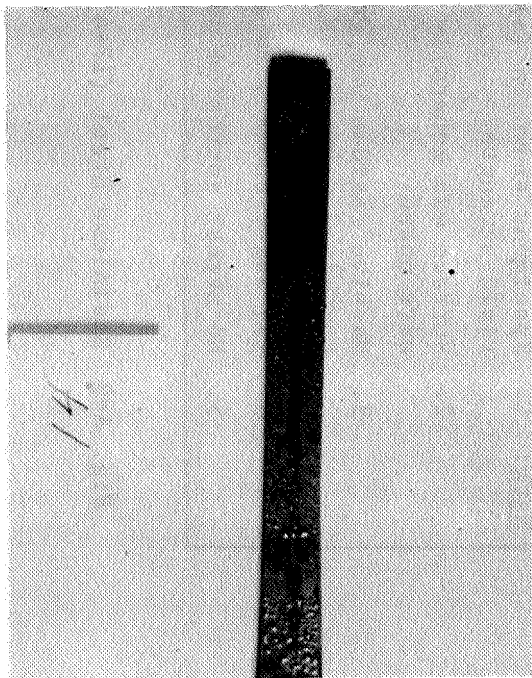
(a) As Removed from Test



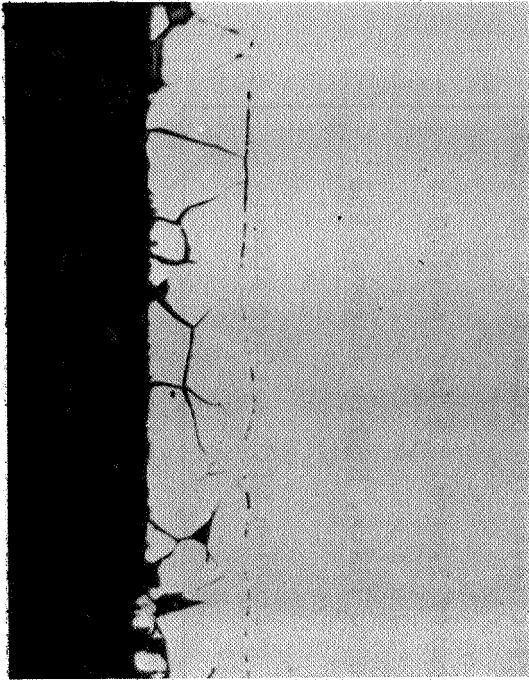
(b) Specimen Section (200X)

- Note:
1. Specimens shown in (a) above were stressed to 50% and 75% of their yield strength.
 2. Surface was oxidized and pitted.
 3. Massive residue was green and brown to black, highly viscous and contained entrapped N_2O_4 .
 4. (b) above shows that pitting and intergranular attack occurred. Depth of intergranular corrosion was approximately 0.002 in.
 5. No stress corrosion occurred.

Fig. A-7 Stainless Steel (Type 347) after Exposure to N_2O_4 for 600 hr at 275°F



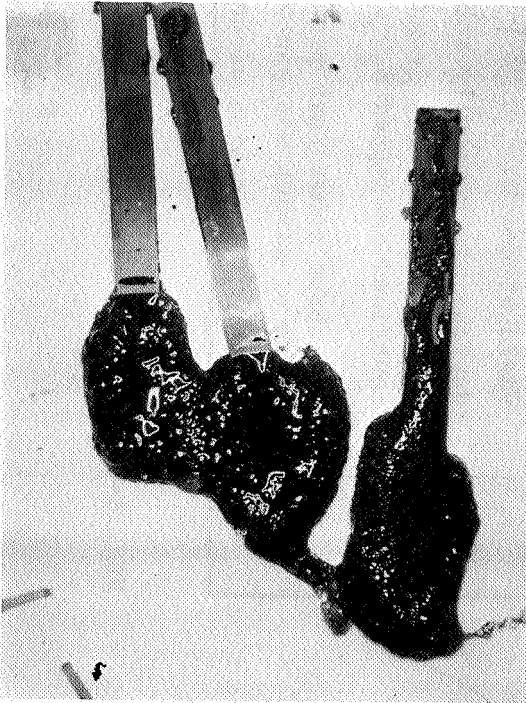
(a) As Removed from Test



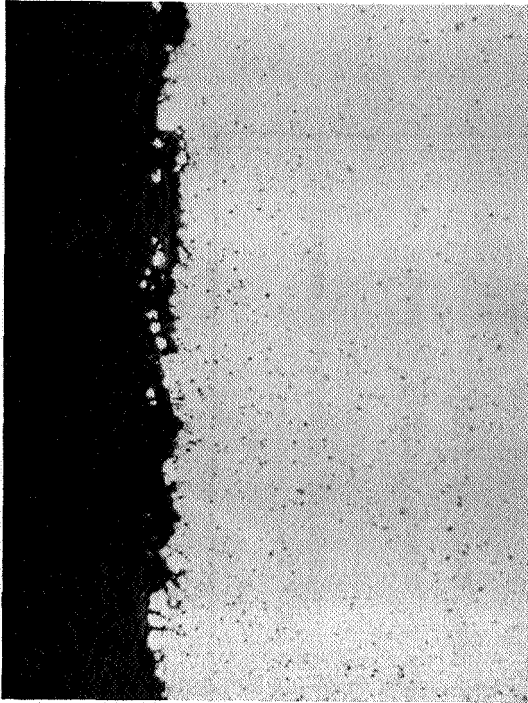
(b) Specimen Section (200X)

- Note:
1. Specimens shown in (a) above were stressed to 50% and 75% of their yield strength.
 2. Surface was generally etched with some deep pitting.
 3. Massive residue was green and brown to black, highly viscous and contained entrapped N_2O_4 .
 4. (b) above shows that pitting and intergranular corrosion occurred. Depth of attack was approximately 0.002 in.
 5. No stress corrosion occurred.

Fig. A-8 Stainless Steel (Type 304) after Exposure to N_2O_4 for 600 hr at 275°F



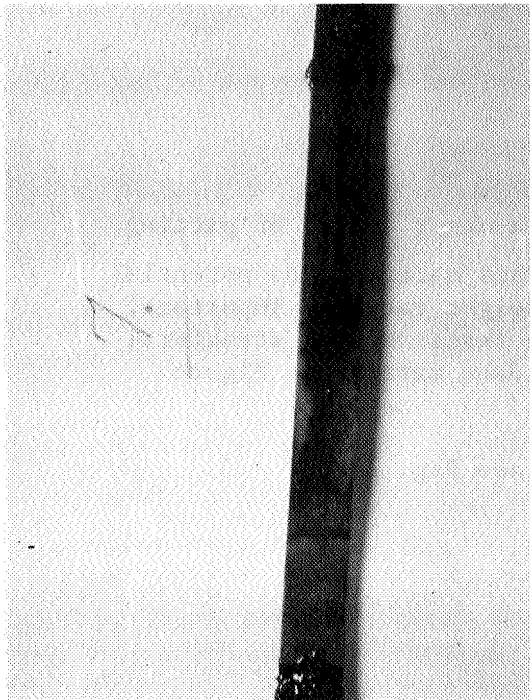
(a) As Removed from Test



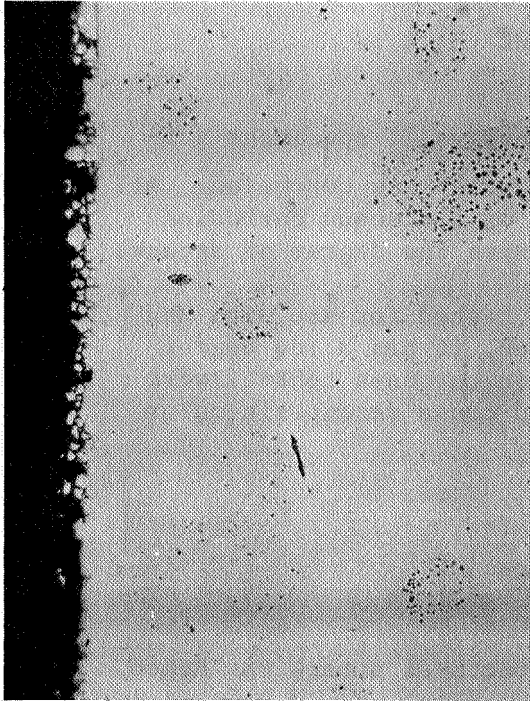
(b) Specimen Section (200X)

- Note:**
1. Specimens shown in (a) above were prestressed to 50% and 75% of their yield strength.
 2. Surface was generally etched to a matte finish. A few small pits were noted.
 3. Massive residue was green and brown to black, highly viscous, and contained entrapped N_2O_4 .
 4. (b) above shows some intergranular corrosion with surface attack.
 5. No stress corrosion occurred.

Fig. A-9 Stainless Steel (17-4PH) after Exposure to N_2O_4 for 600 hr at 275°F



(a) As Removed from Test



(b) Specimen Section (200X)

- Note:**
1. Specimens were stressed to 50% and 75% of their yield strength.
 2. Surface was oxidized and pitted and covered with large quantities of a green to brown viscous deposit.
 3. (b) above shows that pitting and light intergranular corrosion occurred. No stress cracking was observed.

Fig. A-10 Stainless Steel (17-7PH) after 600 hr Exposure to N_2O_4 at 275°F

D. MARAGING STEEL

N₂O₄ Exposure - Severe attack sustained. All specimens suffered pitting, intergranular corrosion, and stress corrosion. All specimens were broken. Large quantities of a viscous corrosion product were found. Figure A-11 illustrates the degree of attack.

MMH Exposure - This material was not tested for use with MMH because, unlike other materials considered, it has a low degree of resistance to rusting. If manufacturing process control were not perfect, this type of corrosion could cause decomposition or possibly ignition of the fuel.

E. CARPENTER 20Cb

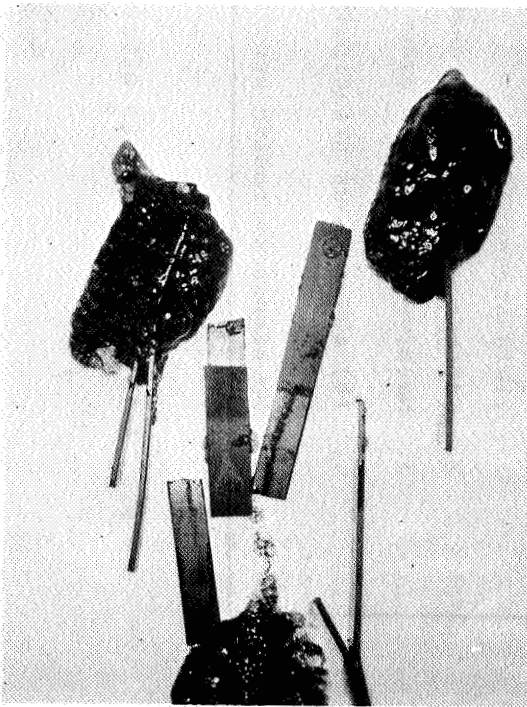
N₂O₄ Exposure - A minor amount of structural attack was noted. Corrosion products were formed that would impair engine performance. Figure A-12 illustrates the degree of attack sustained.

MMH Exposure - No attack occurred (see Fig. A-23).

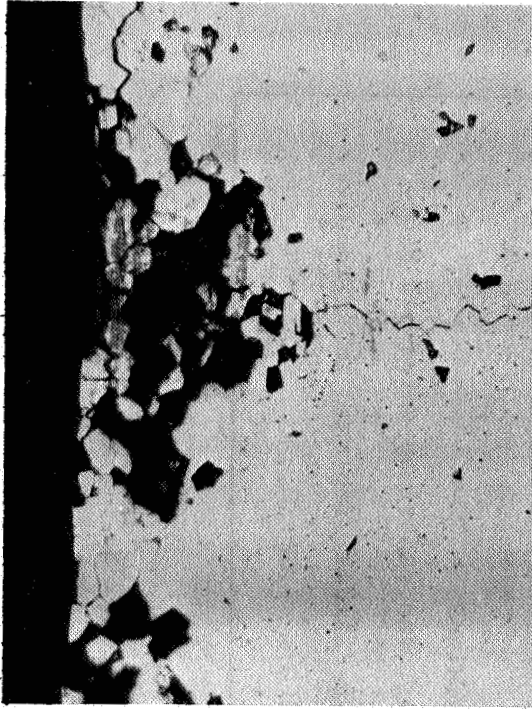
F. HASTELLOY C

N₂O₄ Exposure - Light corrosion and a minor amount of pitting occurred. Specimens were discolored. This material has potential application except where a strength-to-weight ratio is important. It does not present sufficient corrosion resistance or structural properties to be considered a good candidate material. Figure A-13 illustrates the degree of attack.

MMH Exposure - No attack was noted (see Fig. A-23).



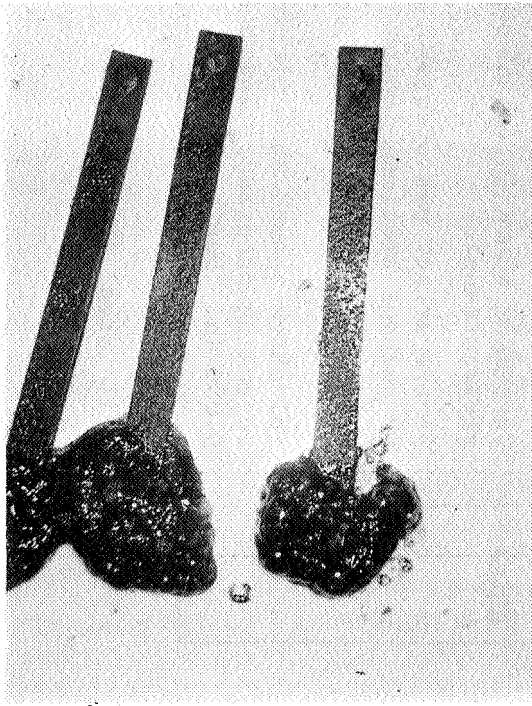
(a) As Removed from Test



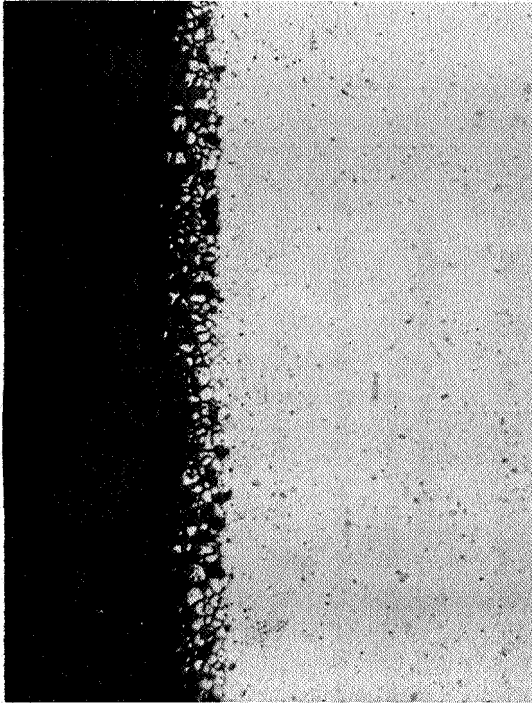
(b) Specimen Section (200X)

- Note:**
1. Specimens shown in (a) above were prestressed to 50% and 75% of their yield strength.
 2. Severe attack was observed on all surfaces. Cracks were numerous throughout specimen length. All specimens were broken.
 3. Massive residue was dark brown, viscous, and contained entrapped N_2O_4 .
 4. (b) above showed that pitting, intergranular corrosion, and stress corrosion occurred. Depth of intergranular corrosion was approximately 0.006 in. Depth of stress corrosion was greater than 0.012 in.

Fig. A-11 Steel (Maraging) after Exposure to N_2O_4 for 600 hr at 275°F



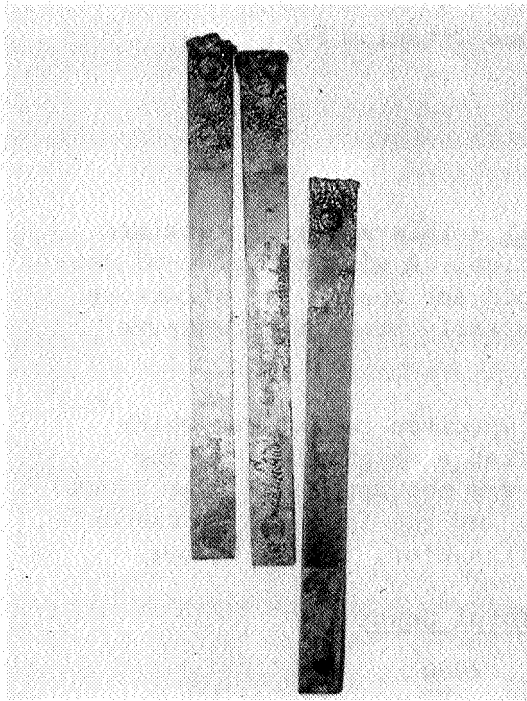
(a) As Removed from Test



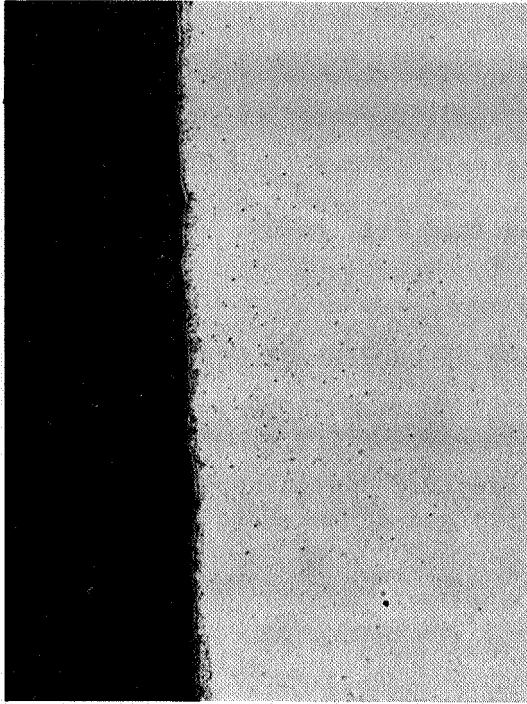
(b) Specimen Section (200X)

- Note:
1. Specimens shown in (a) above were prestressed to 50% and 75% of their yield strength.
 2. Surface was heavily etched and oxidized
 3. Massive residue was green, viscous, and contained entrapped N_2O_4
 4. (b) above shows principally surface attack with some intergranular corrosion.
 5. No stress corrosion occurred

Fig. A-12 Carpenter 20 Cb after Exposure to N_2O_4 for 600 hr at 275°F



(a) As Removed from Test



(b) Specimen Section (200X)

- Note:**
1. Specimens shown in (a) above were prestressed to 50% and 75% of their yield strength.
 2. Surface was pitted and covered with a brown stain. Residue at ends were result of attach on Inconel rivets.
 3. (b) above shows evidence of light pitting and intergranular corrosion.
 4. No stress corrosion occurred.

Fig. A-13 Hastelloy C after Exposure to N_2O_4 for 600 hr at $275^\circ F$

G. A-286

N₂O₄ Exposure - Pitting and intergranular corrosion occurred. Large quantities of corrosion products were formed (see Fig. A-14).

MMH Exposure - No attack was noted (see Fig. A-23).

H. MATERIALS FOR POSITIVE DISPLACEMENT OF PROPELLANT UNDER ZERO-GRAVITY CONDITIONS

This section is separate since a specific design application is involved. It discusses both metallic and nonmetallic materials.

1. N₂O₄ Exposure

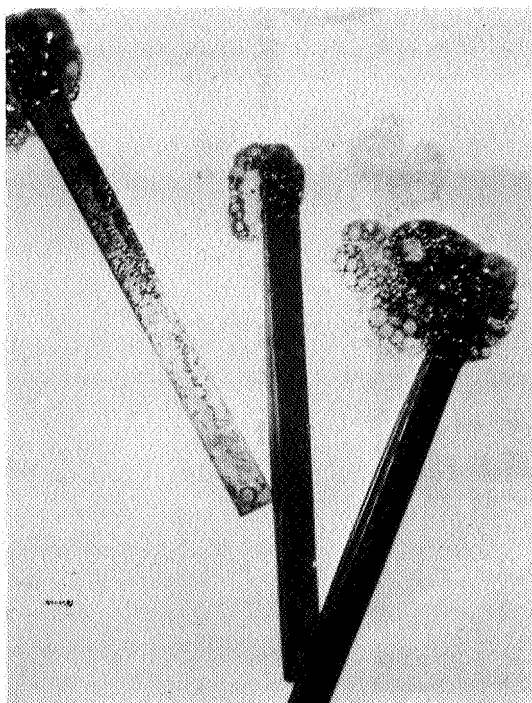
Materials tested included:

Teflon (TFE/FEP Laminate)	Aluminum Screens (6061 and 5056)
Butyl Rubber	Silastic Rubber
Ethylene Propylene Rubber	Etched Titanium Foil Kynar
Nickle Screen	
Nitroso Rubber	

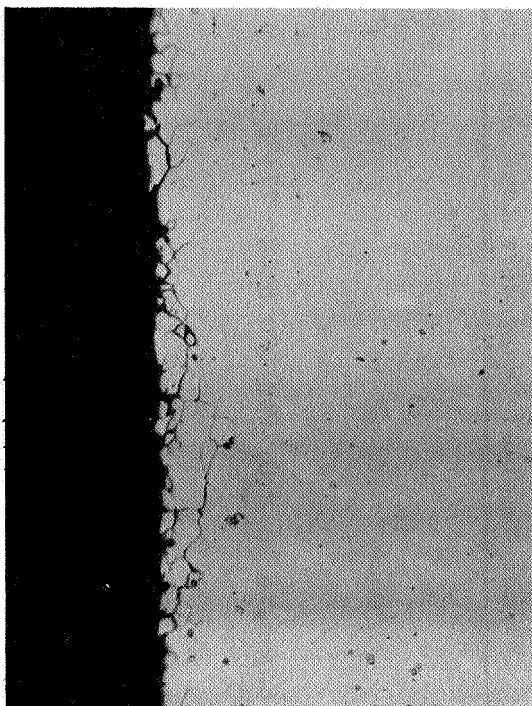
Teflon Laminate (bladder material) - This material retained more than 80% of its physical properties. A slight degree of surface attack was noted. This attack did not change the structure of material removed, instead, it apparently broke the cohesive bond of the sintered material.

A white flock was visible in the specimen containers which were of such inconsequential weight that a weight loss of the specimen (measured to 0.1 milligram) was not apparent. The flock was subjected to millipore filtration and the filtrate microscopically examined for particle size distribution, listed below:

<u>Range (microns)</u>	<u>Total Count</u>
25 to 100	2000
100 to 300	127
300 to 500	25
Over 500	5



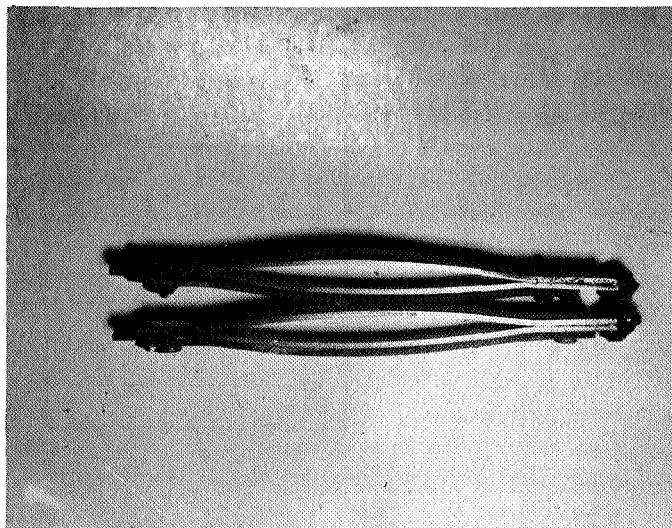
(a) As Removed from Test



(b) Specimen Section

- Note:**
1. Specimens shown in (a) above were stressed to 50% and 75% of their yield strength.
 2. Surface was oxidized and pitted and covered with large quantities of a green to brown viscous residue.
 3. (b) above shows that pitting and intergranular corrosion occurred. Depth of corrosion was approximately 0.002 in.
 4. No stress corrosion was noted.

Fig. A-14 A-286 after Exposure to N_2O_4 for 600 hr at 275°F



Note: No indication of a significant change in compatibility for any individual material was noted when a bimetallic specimen was tested. When a material that produced the viscous residue was in contact with a material that did not produce this material, surface attack was slightly more severe. The only exception was 6Al-4V titanium which was unaffected in all instances. Photographs of the bimetallics are not included since results of individual material tests were representative.

Fig. A-15 Bimetallic Couples after Exposure to N_2O_4 for 600 hr at 275°F

Maximum particle size was 650 microns. This flock is not considered to be detrimental to the propulsion system for the following reasons :

- 1) Particle size is deceptive because sufficient cohesive forces exist to cause many small particles of Teflon to cohere and present the effect of a large particle;
- 2) Teflon particles of this type are easily extruded through a filter or injection plate.

Aluminum Screens - Aluminum screens were pitted, lost section thickness, and were partially clogged by corrosion products (see Fig. A-16).

Butyl Rubber - The rubber ignited after 12 minutes.

Ethylene Propylene Rubber - This rubber lost all measurable physical properties and experienced excessive swelling.

Silastic Rubber - This rubber dissolved.

Etched Titanium Foil - No significant attack occurred on this foil. This material would be usable providing it could be manufactured with a thickness sufficient to satisfy its structural application (see Fig. A-17).

Nickle Screen - The nickel screen was severely attacked with a resultant heavy deposit of nickle nitrate (see Fig. A-18).

Kynar - The Kynar was severely attacked. It is of no value for this application.

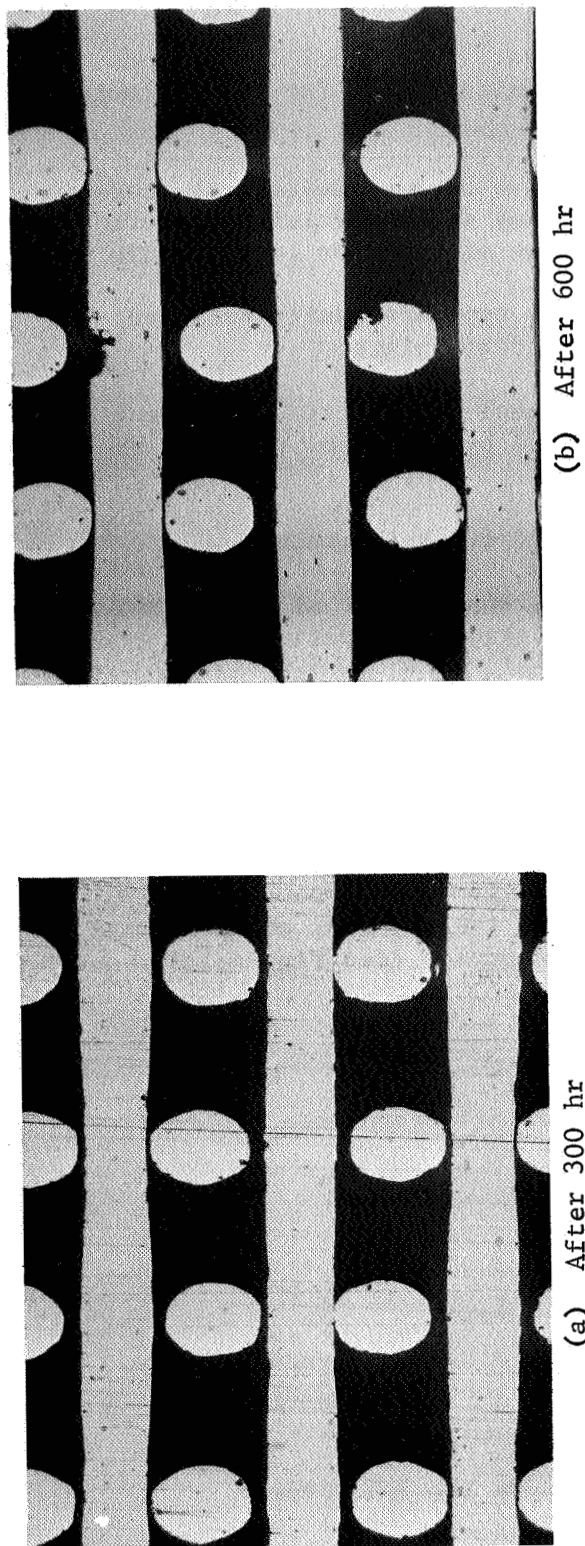
Nitroso Rubber - This rubber lost all measurable properties.

2. MMH Exposure

Materials tested included:

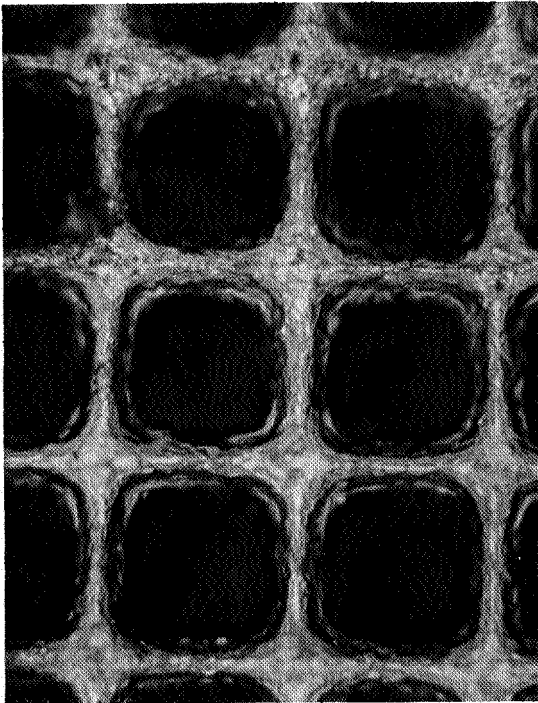
304 Stainless Steel Screen	Kynar
Teflon (TFE/FEB Laminate)	Aluminum Screen (6061 and 5056)

304 Screen - There was no attack on the 304 screen.

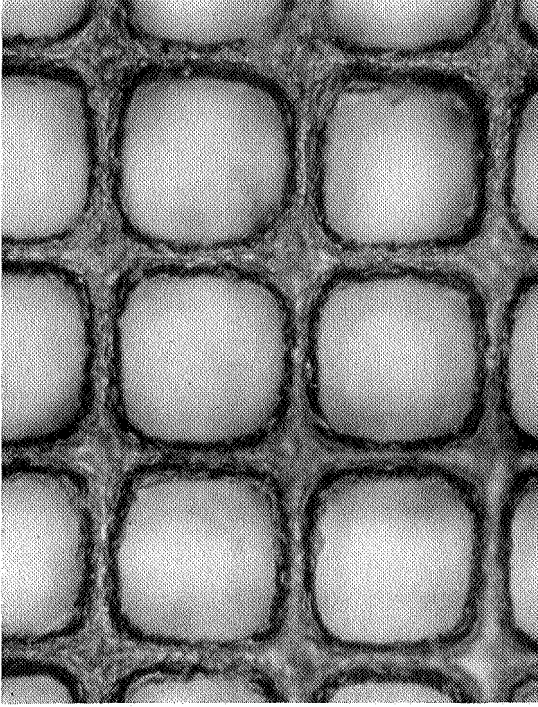


- Note:
1. Screens sustained a general surface attack with deep pitting in some areas.
 2. Screen was coated with a white corrosion product.

Fig. A-16 Aluminum Screens (5056) after Exposure to N_2O_4 at $275^\circ F$



(a) Before Exposure to N_2O_4



(b) After 600 hr at 275°F (200X)

Note: 1. No indication of etching, pitting, or intergranular corrosion was found through surface and microsection examination.

2. No weight loss was noted when sample was weighed to 0.1 milligram.

3. Variation of appearance of the foil was the result of lighting during photographing, not chemical attack.

Fig. A-17 Etched Titanium Foil after Exposure to N_2O_4 for 600 hr at 275°F

Note: Formation of massive quantities of green nickel nitrate along with severe pitting of the wire was noted.

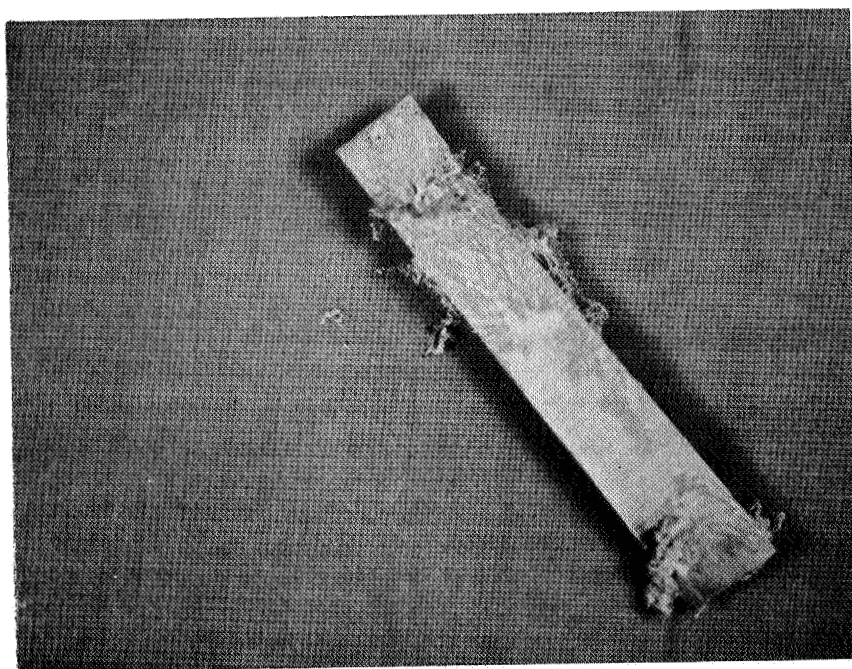


Fig. A-18 Pure Nickel Screen after Exposure to N_2O_4 for 600 hr at 275°F

Kynar - The Kynar was severely attacked. **It** is of no value for this application.

Teflon (TFE/FEP) - The Teflon suffered no **attack**.

Aluminum screens - No attack occurred on the aluminum screens.

I. NONMETALS NOT IN CONTACT WITH PROPELLANTS DURING TERMINAL DRY HEAT STERILIZATION CYCLES

1. Adhesives

Items tested included:

Dow-Corning 93-046 Silicone Adhesive

Armstrong A-6 Epoxy Adhesive

Hysol 1-C Epoxy Adhesive

Aluminum Filled Epoxy Adhesive

See Table A-1 for physical properties.

2. Plastics and Rubber Sheet Film

Items tested included:

Kynar

Teflon (TFE and FEP)

SR 634 Butyl

S-9711 Silicone Rubber

Aluminized Mylar

EPR-1

Silicone Rubber A-60

See Table A-2 for physical properties

Table A-1 Properties of Adhesives after Exposure to 275°F

Material	Tensile Shear Adhesion (average psi)			Mode of Failure
	Control	300 hr	600 hr	
Dow-Corning 93-046	160	130	227	80% adhesive; 20% cohesive
Hysol 1-C Epoxy	1000	2280	2440	85% adhesive; 15% cohesive
Armstrong A-6 Epoxy	780	3090	2376	10% adhesive; 90% cohesive
Devcon F Epoxy	530	2360	3910	2% adhesive; 98% cohesive
<u>Note:</u> A significant increase in shear strength is shown for each adhesive after exposure to 275°F. This is a result of further cross linking of molecules, which is attendant with post cures for these types of materials. Along with the increase in shear strength, a decrease in flexibility occurs however, which causes the adhesive to become brittle and lose its ability to resist failure under vibrational loading.				

3. Potting Encapsulating and Sealing Resins

Items tested included:

Epon 828 - Mica Filled (amine cure)

PR-1527 Polyurethane Casting Resin

RTV 602 Silicone Potting Resin

Dow-Corning 4-9-0031 Silicone Resin (specified in Martin Marietta Materials Specification MMS M138)

Epon 828/Versamide 140 Potting Resin

Martin MMS M138 Silicone Sealant (Dow-Corning)

See Table A-3 for physical properties.

4. Coatings and Finishes

Items tested included:

Martin Marietta MMS K227 White Acrylic Missile Lacquer

Martin Marietta MMS K456 Ablative Coating

Martin Marietta MMS K474 High Emissivity Silicone Coating

MMS K227

Control: Excellent adhesion. No flaking with checker-board cut and tape test. Good adhesion after bending 180 deg around 1/4-in. mandrel.

Test: Embrittled, adhesion fair to good. Flakes away with checkerboard cut. Loses adhesion in the bend area after 180 deg bend around a 1/4-in. mandrel (300 hr test).

Test: Embrittled, adhesion fair. Flakes away with checker-board cut. Loses adhesion in the bend area after 180 deg bend around a 1/4-in. mandrel. Specimen yellowed significantly (600 hr test).

MMS K456

- Control: Excellent adhesion, typically soft. Failed 180 deg bend on 1/4-in. mandrel due to coating thickness. Passed 90 deg bend on 1-in. mandrel.
- Test: Excellent adhesion. Tougher and darker than control specimens in cross section. Failed 90 deg bend over 1/4-in. and 1-in. mandrels (300 hr test).
- Test: Excellent adhesion. Tougher and darker than control specimens in cross section, Failed 90 deg bend over 1/4-in. and 1-in. mandrels (600 hr test).

MMS K474

- Control: Good adhesion. Coating normally low strength. Passed 180 deg bend over 1/4-in. mandrel. No flaking.
- Test: Good adhesion. Coating somewhat stronger than control sample. Passed 180 deg bend over 1/2-in. mandrel. No flaking (300 hr test).
- Test: Good adhesion. Coating somewhat stronger than control sample. Passed 180 deg bend over 1/2-in. mandrel. No flaking. The coating yellowed significantly (600 hr test).

Solar Absorptivity and Emissivity

<u>Material</u>	<u>Control</u>		<u>300 hr</u>		<u>600 hr</u>	
	<u>Absorb</u>	<u>Emiss</u>	<u>Absorb</u>	<u>Emiss</u>	<u>Absorb</u>	<u>Emiss</u>
MMS K227	0.22	0.85	0.28	0.86	0.36	0.86
MMS K474	0.14	0.86	0.18	0.84	0.16	0.85

J. REACTIVITY OF ETHYLENE OXIDE/12%, FREON 12/88% WITH PROPELLANT LEAKS

MMH was injected into a test chamber containing the decontaminating gas (maintained at 22 psia at 122°F) in sufficient quantity to produce a concentration of 5×10^5 ppm. A 6-psi pressure rise resulted with a temperature increase less than 5°F.

N_2O_4 was injected into the same atmosphere resulting in a 26-psi pressure rise and a 22°F temperature increase. A low-grade reaction apparently occurred; however, the pressure increase could result in rupture of the decontamination chamber if proper detection and purge systems are not incorporated.

K. CAPABILITY OF ASI PROPELLANT DETECTION UNITS TO OPERATE IN AN ATMOSPHERE OF THE DECONTAMINANT GAS

The fuel cell reacted erratically when exposed to the sterilant gaseous atmosphere. It was not found suitable in this application.

The oxidizer cell performed normally and satisfactory for use.

L. PASSIVATION OF MMH SYSTEMS

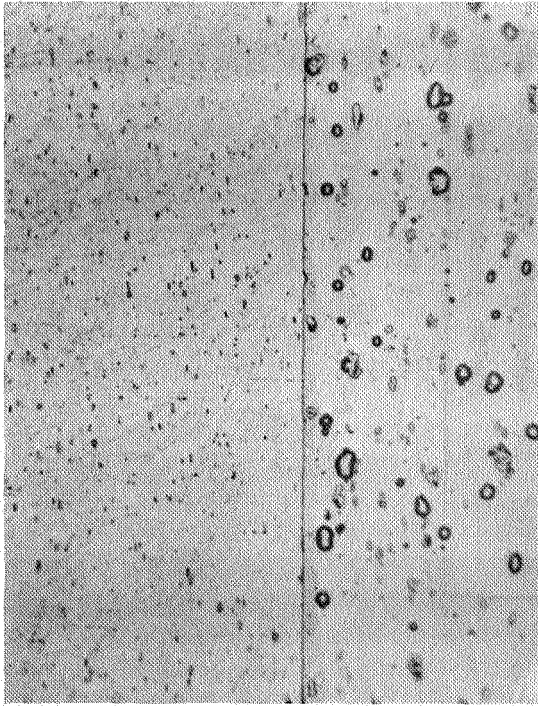
All surfaces that contact MMH require both cleaning and passivation with either a MMH/water mixture or pure fuel, both operated at elevated temperatures. When any alloy tested was exposed to heated MMH without proper chemical cleaning, decomposition was observed. This was evident through release of gas evolution resulting in unusually high pressure in the test vessels. When all materials tested were chemically cleaned and passivated and loaded into the 600-hr terminal dry heat test chamber, no unusual pressure increases were observed.

Propellant samples were taken from a glass container with no metal (Test Sample 1) sample present and from another vessel containing a dissimilar combination of metals (Test Sample 2). This sample consisted of a bimetallic specimen of 2014 aluminum and 304 stainless steel joined with Monel rivets.

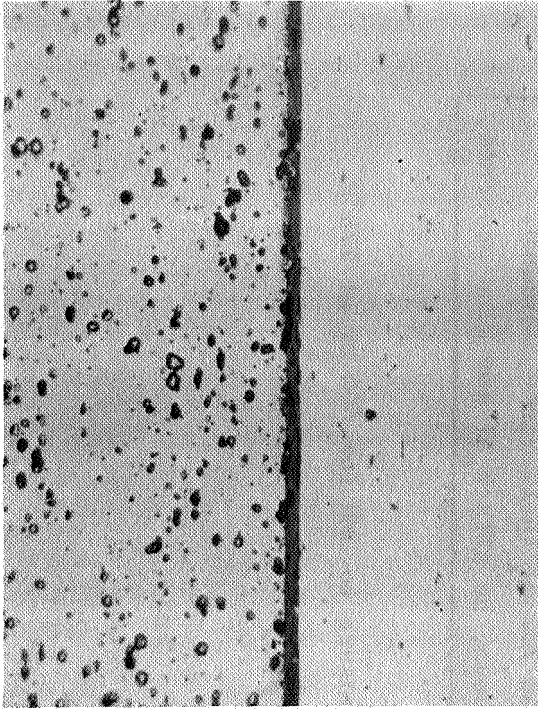
Materials selected were considered of the most potential reactive nature based on previous data from nonpassivated tests.

The propellant decomposition is listed in the following tabulation (based on gas chromatograph results):

	<u>Standard MMH</u>	<u>Test Sample 1</u>	<u>Test Sample 2</u>
MMH	99.49%	98.06%	97.61%
Water	0.46	1.48	1.94
Hydrazine	0.00	0.22	0.20
Ammonia	0.04	0.23	0.24
Air	0.01	0.01	0.01



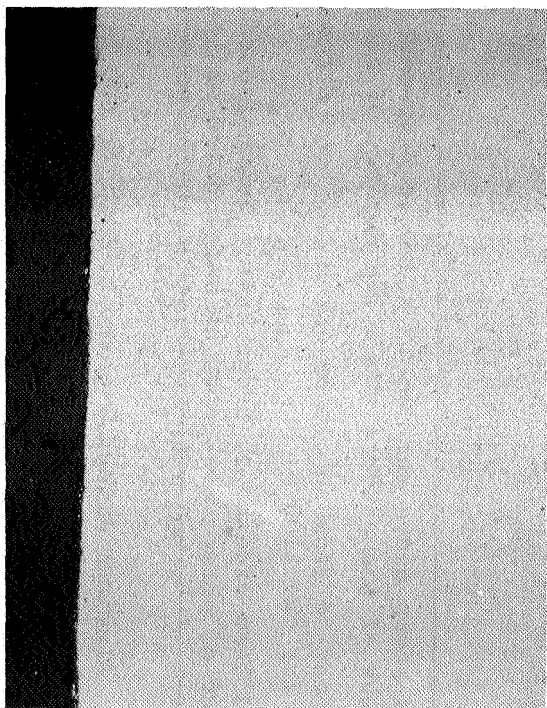
(a) Top Microsection 1100-0 (200X)
Bottom Microsection 2014-T6 (200X)



(b) Top Microsection 2219-T8 (200X)
Bottom Microsection 6061-T6 (200X)

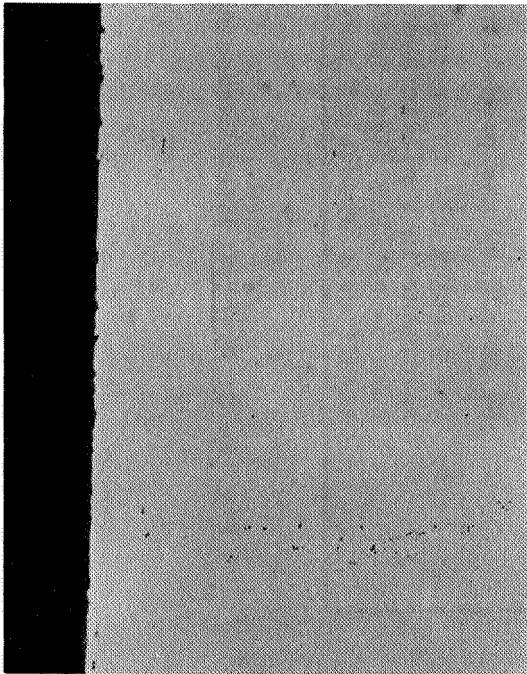
- Note:**
1. Photomicrographs shown above are double pictures representing alloys indicated. The line at the center of the picture shows the surface of each specimen.
 2. Sections were obtained from specimens which had been stressed to 75% of their yield strength except for the 1100-0.
 3. **All** sections had been exposed to propellant. No unexposed samples are presented since no evidence of attack was noted on examination of specimens.

Fig. A-19 Aluminum Alloys Exposed to **MH** for 600 hr at 275°F

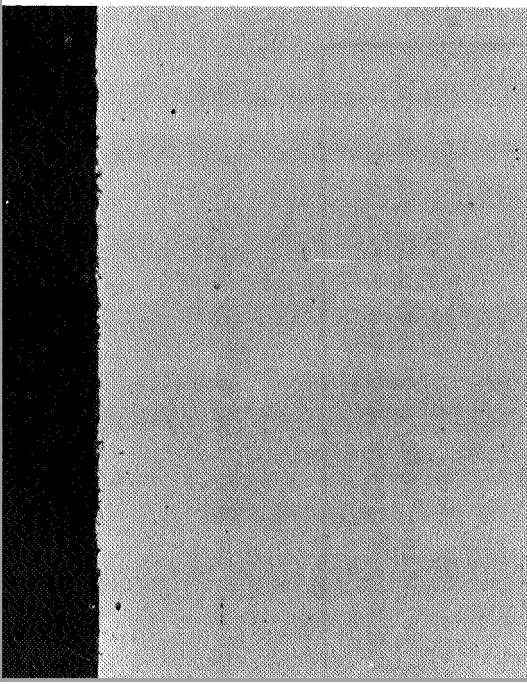


Note: 1. Specimens were stressed to 50 and 75% of their yield strength.
2. As is shown in the photograph above, no attack of any kind occurred.

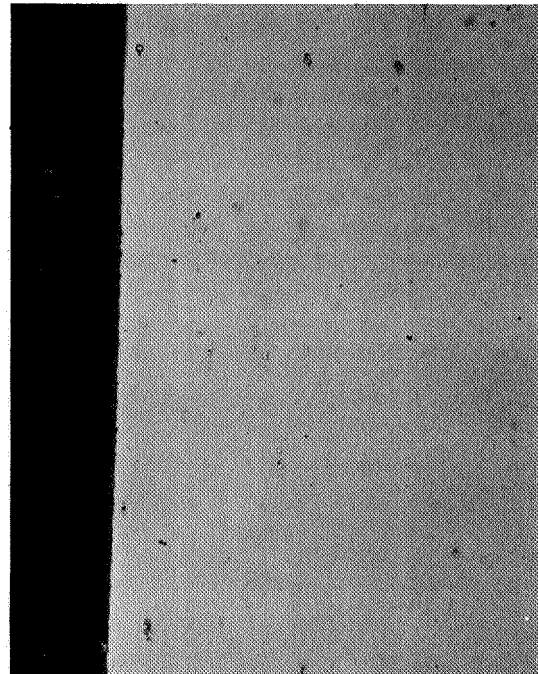
Fig. A-20 Titanium (6Al-4V) after Exposure to MMH for 600 hr at 275°F



(a) 304 Stainless Steel (200X)



(b) 321 Stainless Steel (200X)

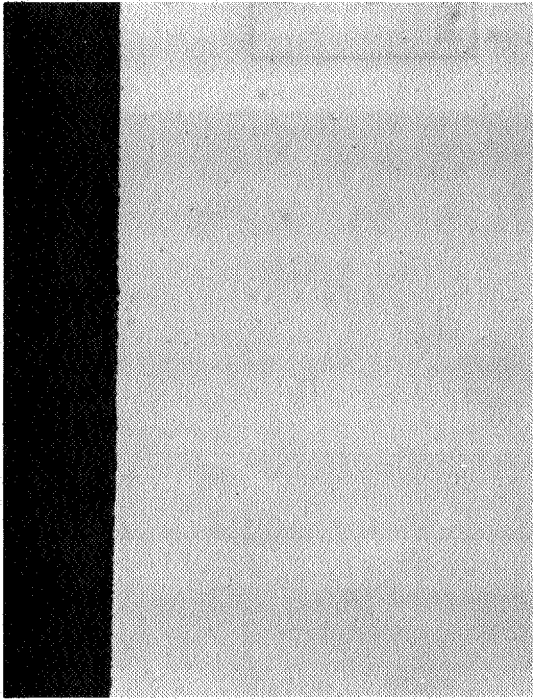


(c) 347 Stainless Steel (200X)

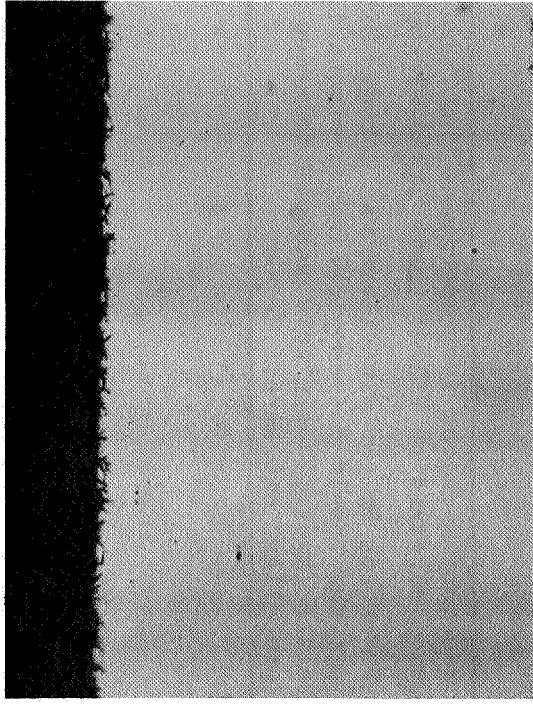
Note:

1. Sections were cut from specimens that had been stressed to 75% of their yield strength,
2. No attack by the propellant was noted. Minor pitting resulted from pickling specimens in a nitric/hydrofluoric acid aqueous solution before propellant exposure.

Fig, A-21 300 Series Stainless Steels Exposed to MMH for 600 hr at 275°F



(a) 17-4PH Stainless Steel (200X)

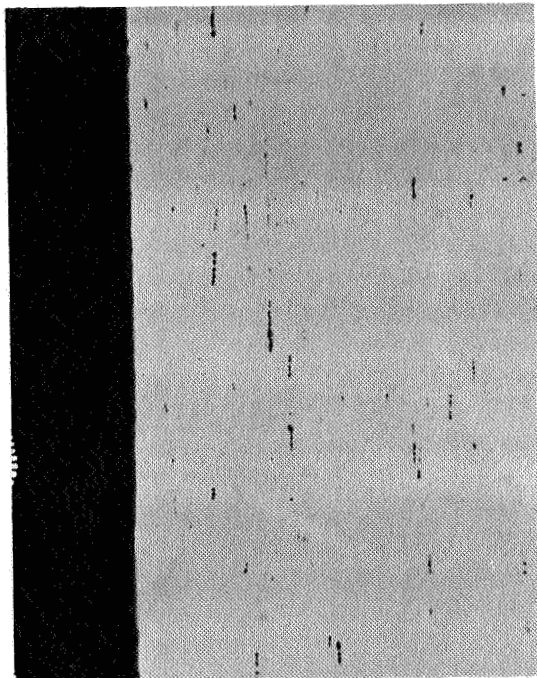


(b) 17-7PH Stainless Steel (200X)

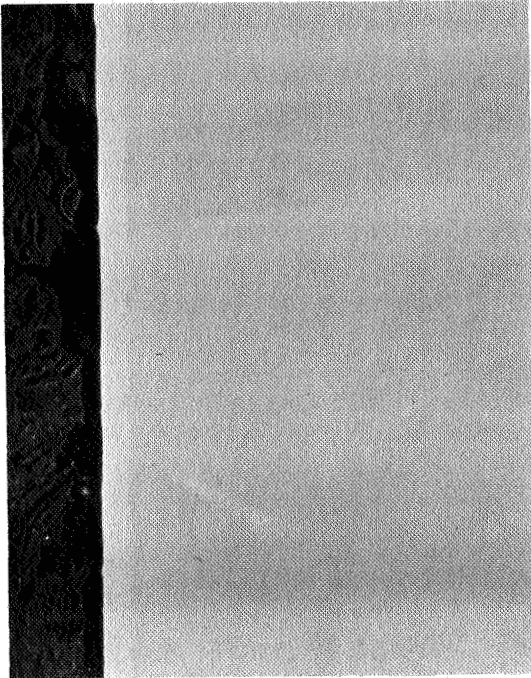
Note:

1. Sections were cut from specimens which were stressed to 75% of their yield strength.
2. No attack by the propellant was noted.
3. The pitting and intergranular corrosion evident in (b) above resulted from pickling specimens in a nitric/hydrofluoric acid aqueous solution before propellant exposure. This particular alloy had been heat treated in an air furnace and had sustained a significant amount of surface oxidation.

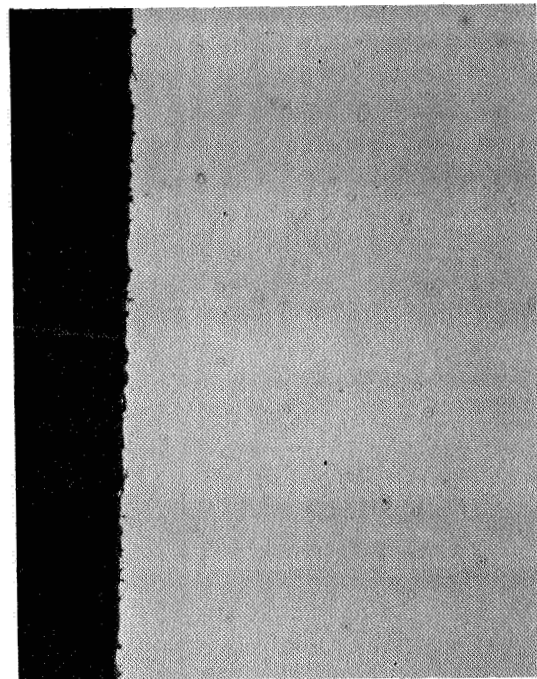
Fig, A-22 Precipitation Hardening Stainless Steels Exposed to MMH for 600 hr at 275°F



(a) Carpenter 20-Cb (200X)



(b) Hastelloy C (200X)



(c) A-286 (200X)

Note: 1. Sections were cut from specimens that had been stressed to 75% of their yield strength.
2. No attack by the propellant was noted.

Fig A-23 Special Alloys Exposed to for 600 hr at 275 F

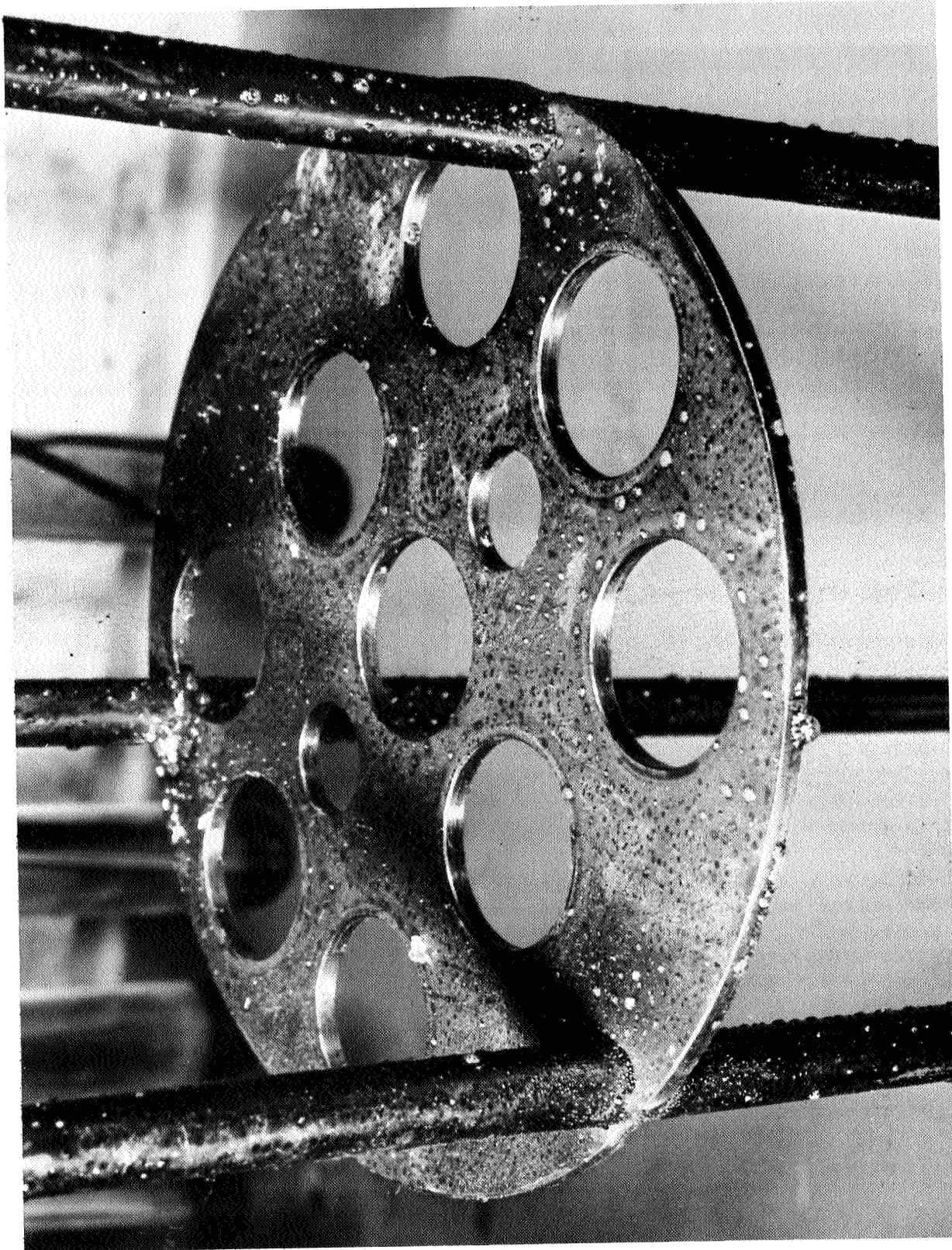


Fig. A-24 Oxidizer Bomb Rack

V. PROCEDURES

A variety of test procedures were used during this program. These procedures included: prescreening, isolation prescreening, equivalent sterilization cycles, and decontaminating gas - propellant reactions.

All propellant exposures were accomplished using monomethyl hydrazine (MIL-P-27402) and nitrogen tetroxide (MIL-P-26539B). Nitric oxide (NO) was bubbled through the N_2O_4 . The procedures are listed in the following sections.

A. PRESCREENING TESTS

The materials were exposed to oxidizer at sterilization temperature by inserting specimens in 300 series stainless steel Hoke cylinders that were cleaned in accordance with Martin Marietta Drawing 327-9020000, for liquid oxygen use. Test cylinders were loaded with propellant and stored in an oven at 275°F for duration of exposure. Throughout testing pressure and temperature were monitored using:

- 1) Pressure - Tabor Model 176 transducer; 0-1000 psi range with Tabor indicator (Model 236) for pressure readout;
- 2) Temperature - Chromel/Alumel thermocouple (closed end) with Leeds and Northrup Potentiometer (Model 1214006).

The materials were exposed to fuel at sterilization temperature by inserting specimens in a 300 series stainless steel flared tubing assembly that had been cleaned in accordance with Martin Marietta Drawing 327-9020000, for liquid oxygen use. Tubing assembly was then loaded with propellant and stored in an ethylene glycol bath for the duration of exposure. Throughout testing pressure and temperature was monitored using:

- 1) For pressure - Tabor Model 176 transducer; 0-150 psi range with Tabor indicator (Model 236) for pressure;
- 2) For temperature - Chromel/Alumel thermocouple (closed end) with Leeds and Northrup Potentiometer (Model 1214006).

B. ISOLATION PRESCREENING TESTS

Formation of corrosion products, when samples were exposed to N_2O_4 in the 300 series stainless steel container, clouded results since the containers were attacked. As a result, a series of tests were conducted with the test chamber of a similar family or the same alloy e.g., 2014-T6 was tested in a 6061-T6 vessel or a 6061-T6 specimen tested in a 6061-T6 vessel. All other conditions were the same as those defined in prescreening tests described previously.

C. EQUIVALENT STERILIZATION CYCLE TEST (SCREENING)

Screening tests exposed candidate materials to a longer duration under heat sterilization environments. These tests included two environmental conditions -- exposure of materials in contact with propellant and exposure of materials to dry heat.

1. Exposure of Materials in Contact with Propellant

This test was performed for all candidates for propulsion tankage and associated plumbing. Test parameters included the following items.

Test specimens were placed in glass containers. Each container received one or more specimen. No mixing of specimen types occurred in any container.

The glass containers were filled to 3/4 volume with propellant and placed in a test bomb. The bomb was sealed and pressurized to operational pressure with nitrogen gas. Temperature was elevated to $275 \pm 3.5^\circ F$ and maintained throughout the duration of the test.

At the initiation of the test, two bombs for each propellant were prepared as described above and were identified as A-F, B-F, A-0, B-0 (F and O referring to fuel and oxidizer, respectively). Bombs labeled A-F and A-0 were removed from test after 300 hr. Bombs labeled B-F and B-0 were removed from test after 600 hr. Figures A-25, A-26, and A-27 show the bomb and associated equipment.

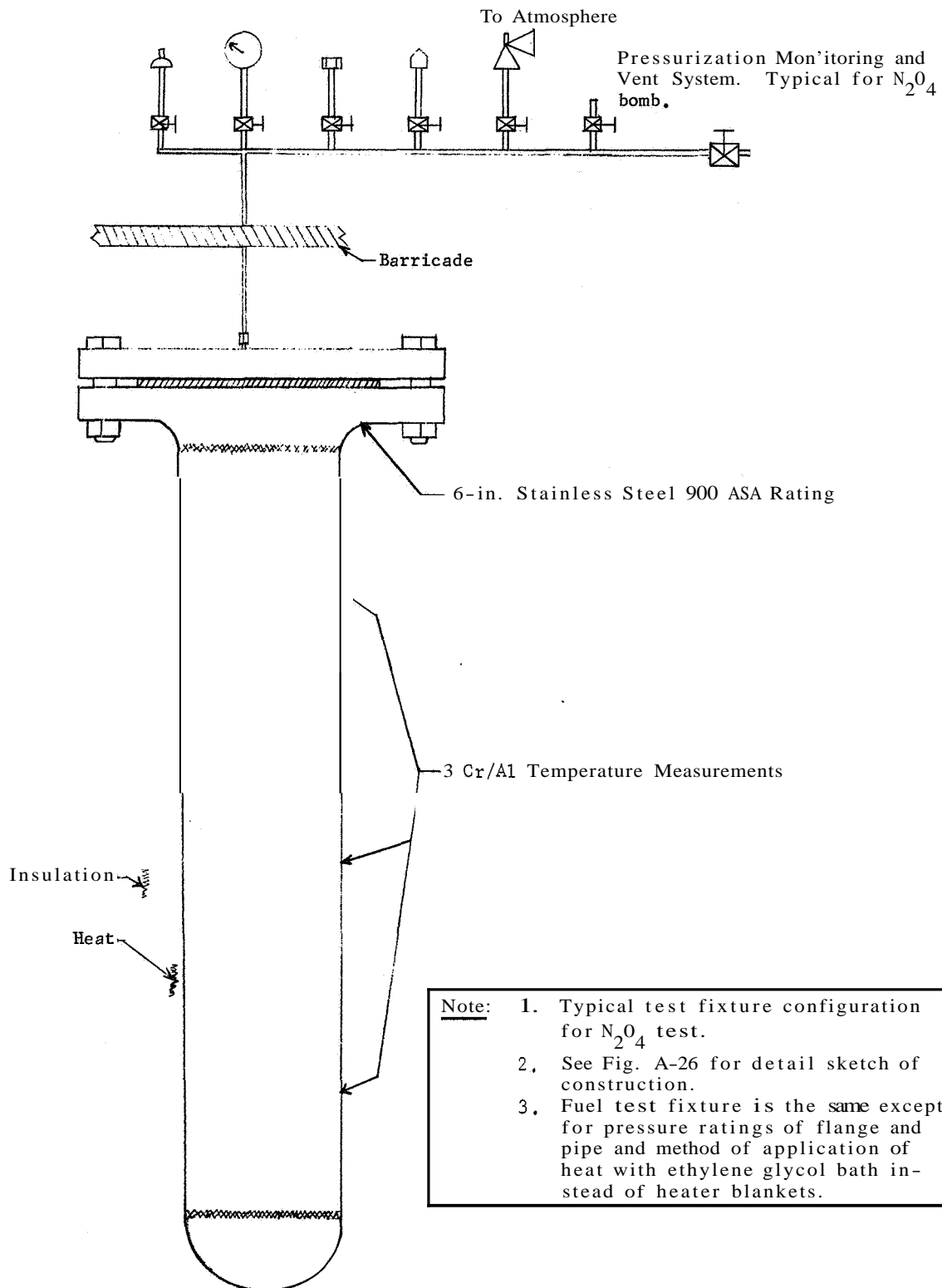
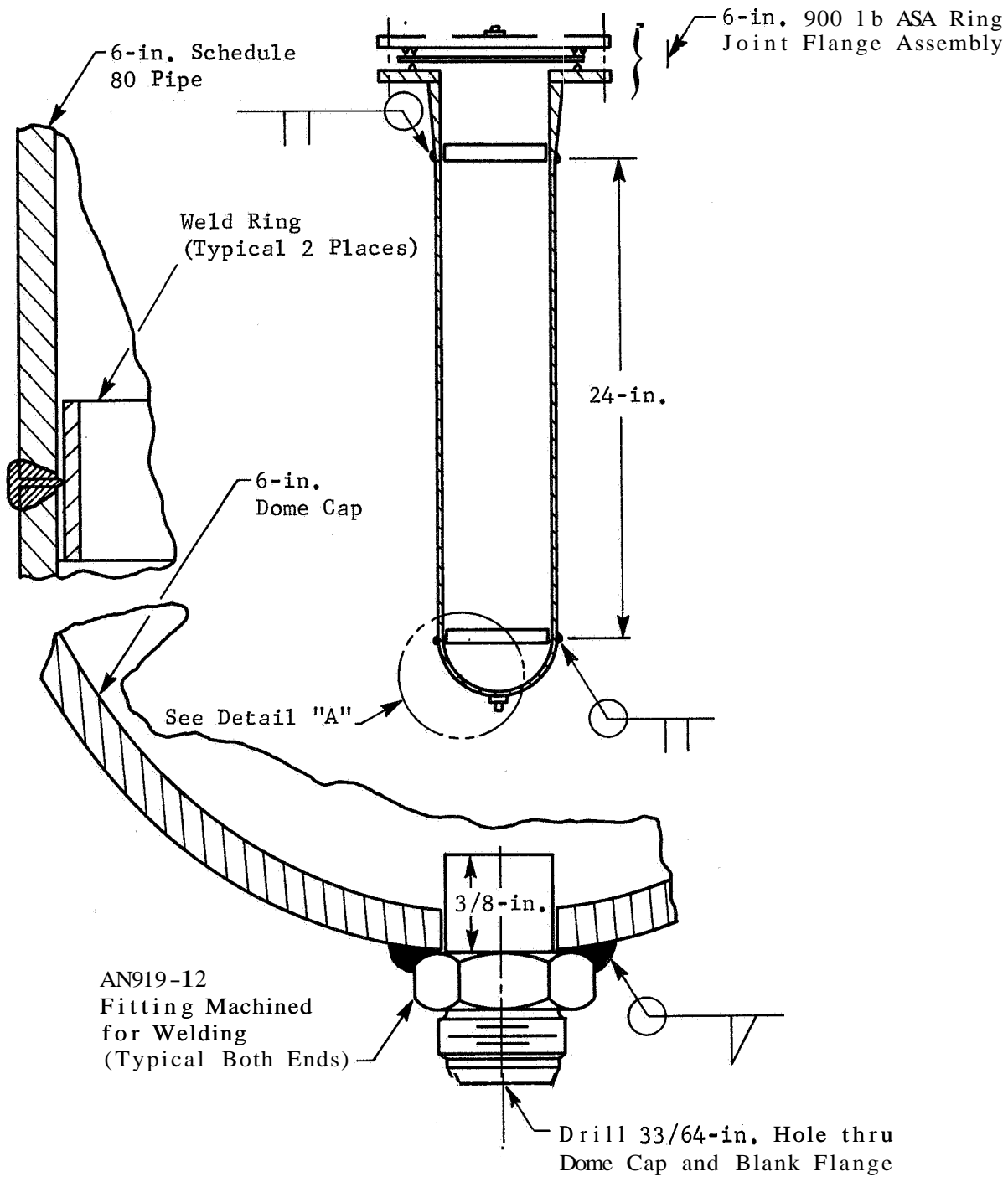


Fig. A-25 Compatibility Screening Test Fixture

Note: Typical for N_2O_4 bomb; fuel bomb is similar except weld rings are not used.



Detail A

Fig. A-26 Test Vessel Details

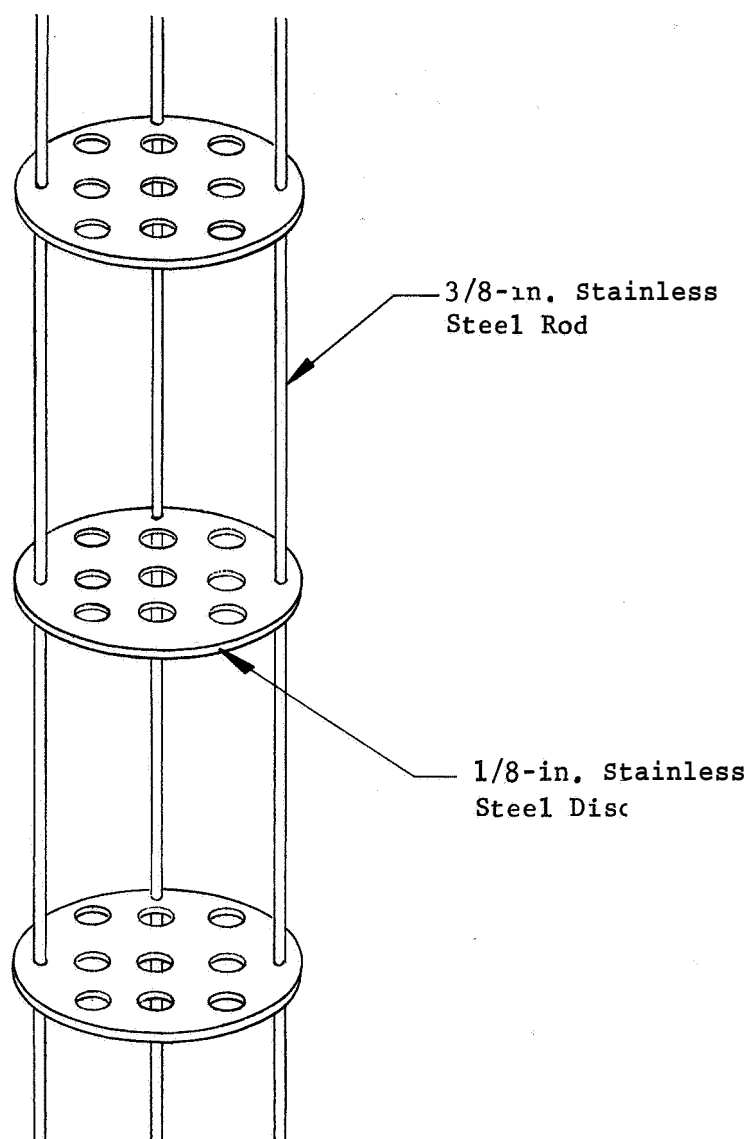


Fig. A-27 Glass Vessel Support Fixture

The standard test fixtures for screening tests were made in accordance with Fig. A-25, A-26, and A-27. Materials of construction were 300 series stainless steel. Specimens were stored on four tiers. Each level accommodated nine glass specimen containers.

The oxidizer test fixture was a seamless cylinder made of schedule 80 - 304 stainless steel, 6 in. diameter and 24 in. long, and a 310 stainless steel flanged ring seal cap. Flange rating was 900 lb ASA. Each oxidizer fixture was hydrostatically tested to 1600 psig with the pressure held for a period of 5 minutes.

The fuel test fixture was constructed in a similar manner to the oxidizer fixture except that schedule 40 pipe wall thickness was used and 150-lb ASA flanges were used. Each fuel fixture was hydrostatically tested at 250 psig with the pressure held for a 5-minute duration.

The fixtures and associated hardware were cleaned according to the requirements of Martin Marietta Drawing 327-9020000, for liquid oxygen use.

Test specimens were contained within the fixture (see Fig. A-2) in glass test vessels. Each pyrex glass vessel was stored in a sealed polyethylene plastic bag until immediately before use. At such time the vessels were handled with tongs and lint-free gloves. Each vessel was cleaned by the following procedures:

- 1) Remove from plastic bag;
- 2) Immerse vessel in an acid solution (1/4 lb of potassium dichromate in one liter of concentrated sulphuric acid) maintained at $80 \pm 5^{\circ}\text{F}$ for 30 minutes minimum;
- 3) Rinse with distilled water;
- 4) Immerse in concentrated nitric acid for a minimum of 10 minutes;
- 5) Rinse with distilled water;
- 6) Immerse in concentrated ammonium hydroxide for 10 minutes minimum (except aluminum alloy);
- 7) Rinse with distilled water;
- 8) Inspect visually;

- 9) Repeat procedures if necessary;
- 10) Rinse with deionized water, check pH of the runoff water and compare to pH of fresh distilled and deionized water. Continue until pH differs by less than ± 0.5 units;
- 11) Blow dry with oil-free nitrogen gas (Robbins filtered);
- 12) Return to sealed plastic bag.

All test containers, racks, and specimens remained sealed in plastic until final assembly. Assembly was performed in the sterilization test area to eliminate the need of moving loaded containers. Individual glass vessels were unwrapped, loaded with specimens and propellant, and placed in the holding rack. The rack was lowered into the metal container a step at a time until it rested on the bottom. The ring seal and flange head were unwrapped and put in place. The head was then bolted in place and attached to the system plumbing. As each container was sealed, it was pressurized with nitrogen and leak checked. A constant gaseous nitrogen purge was maintained on the test fixture during loading operations.

Pressure and temperature measurements were made using Tabor transducer Model 176 and Chromel/Alumel thermocouples. Readout devices consisted of: Sanborn recorder, Honeywell Visicorder, and John Fluke differential voltmeter. Pressure transducers were dead weighted (N_2 static) at 5-psi increments on lower range transducers. Dead-weight psi accuracies are $\pm 0.001\%$ of full scale (Heise gage accuracies). Transducer accuracies include a repeatability of $\pm 0.1\%$ and a linearity of 0.25% full scale with a thermal sensitivity shift of 0.005% of full scale per degree Fahrenheit. Thermocouples were calibrated by selected temperature steps checked against a laboratory thermometer.

Pressure control was maintained by solenoid operated valves, controlled by a pressure switch. Temperature control was governed by a stepless power application to resistive heaters. Using accurate temperature probes, vessel temperature was controlled to $\pm 2^\circ C$ of the required temperature.

2. Exposure of Materials to Dry Heat Cycle

This exposure included candidates for use external to the propellant tank system.

Sterilization temperature effects on physical properties of all nonmetals and metals (that are considered marginal) was evaluated.

Specimens were placed in an oven and maintained at 275°F for 600 hr in a nitrogen atmosphere.

D, REACTIVITY OF DECONTAMINATION ATMOSPHERE WITH PROPELLANTS

Reactivity of N_2O_4 and MMH with the 12% ethylene oxide/88% Freon 12 atmosphere was determined by the following procedure for each propellant:

- 1) Evacuate a 0.4-cu ft stainless steel chamber;
- 2) Heat chamber to 122°F;
- 3) Introduce sufficient water to establish a relative humidity of 45%;
- 4) Pressurize with the decontaminating gas to 12 psig;
- 5) Inject sufficient propellant to establish a fume concentration of 5×10^5 ppm;
- 6) Monitor for temperature and pressure rise.